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FINAL REPORT

FOR

DESIGN AND DEVELOPMENT OF DIBORANE SHIPPING CONTAINER

For Period

7 November 1968 - 18 September 1972

Prepared for
Jet Propulsion Laboratory, California Institute of Technology
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TABLE OF CONTENTS

VOLUME I

Page

INTRODUCTION..... 1

SUMMARY..... 2

DESIGN AND CONSTRUCTION..... 5

- Specifications..... 5
- Selection of Subcontractor..... 5
- Other Uses for Container..... 6
  - Use Container as Feed Tank..... 6
  - Use Container for Oxygen Difluoride..... 6
- Container Design..... 7
  - NASA Recommendations..... 7
  - Unloading Rate..... 8
  - Design Details..... 8
  - Design Approval & Modifications..... 9
- Fabrication and Assembly..... 11
- Container Testing at CVI..... 12
- Container Delivery..... 13
- Modifications..... 13
- Final Drawings..... 13

PERFORMANCE TEST # 1..... 26

- Loading Facility..... 26
- Checkout at Callery..... 26
- Diborane Charge..... 27
- Thermal Test # 1..... 28
- Test Data Analysis..... 29
- Diborane Unloading Tests..... 29

PERFORMANCE TEST # 2..... 39

- Container Modifications..... 39
- Container Testing at CVI..... 39
- Checkout at Callery..... 41
- Repump of Insulation Space..... 41
- Precooling..... 42
- Diborane Charge..... 42
- Thermal Test # 2..... 43
- Test Data Analysis..... 43
- Post-Test Recool..... 45
- Container Cleanout..... 45
- Shipping Test..... 45

TABLE OF CONTENTS (Continued)

Page

DEPARTMENT OF TRANSPORTATION APPROVAL..... 83

Preliminary Contacts..... 83
Design Review..... 83
Request for Special Permit..... 84
Development of Special Permit Terms..... 85
Net Weight Limits..... 85
Test Shipment with Methanol..... 85
Diborane Classification..... 86
Bureau of Explosives..... 87
Special Permit Issue and Amendment..... 87

VOLUME II

APPENDIX A - RECORD OF CONTACTS WITH D.O.T. REGARDING SPECIAL PERMIT FOR 200-POUND DIBORANE SHIPPING CONTAINER

VOLUME III

APPENDIX B - OPERATING INSTRUCTIONS

LIST OF TABLES

Page

1. Specifications for Diborane Shipping Container..... 15
2. Summary of CVI Corporation Proposal..... 18
3. Unloading Test Data Obtained with Methanol..... 31
3A. Analysis of Unloading Test Data..... 31A
4. Container Performance Data - Test # 1..... 32
5. Vacuum Retention Test @ CVI (Nov. 1970)..... 48
6. Thermal Test with LN_2..... 49
7. Diborane Shipping Container Pretest Data..... 50
8. Diborane Shipping Container Precooling..... 59
9. Diborane Charge for 30-Day Storage Test..... 61
10. 30-Day Storage Test Data..... 66
11. 30-Day Storage Test Results
Smoothed Data - Linear Segments..... 71
12. 30-Day Storage Test Results
Smoothed Data - Even Days..... 72
13. Post-Test Recool..... 73

LIST OF FIGURES

Page

1. Preliminary Diagram (CVI Proposal)..... 20
2. Preliminary Flow Schematic (CVI Proposal)..... 21
3. Diborane and Oxygen Difluoride Vapor Pressure..... 22
4. Diborane Shipping Container - Elevation View..... 23
5. Diborane Shipping Container - Plan View..... 24
6. Vacuum Retention Test @ CVI (December 1969)..... 25
7. Loading and Unloading Facility @ Callery..... 34
8. Vacuum Retention Test @ Callery (Dec. 1969 - Jan. 1970)..... 35
9. Container Unloading Test Data with Methanol..... 36
9A. Container Unloading Data..... 36A
10. Performance Data - Test # 1..... 37
11. Continuation Data - Test # 1..... 38
12. Vacuum Retention Tests @ CVI..... 79
13. Repump of Insulation Space Vacuum..... 80
14. Diborane Charge for 30-Day Storage Test..... 81
15. 30-Day Storage Test Results..... 82

INTRODUCTION

Callery Chemical Company has been shipping diborane as a gas since 1953 and as a liquid since 1959. Callery holds DOT Special Permit 930 for shipment of diborane gas and diborane-inert gas mixtures in non-refrigerated overpacks, under specific conditions outlined in the permit. Callery also holds DOT Special Permit 970 for shipment of liquid diborane in overpacks containing dry ice for refrigeration. These permits were re-written and reissued in their present wording in January 1968.

Prior to this program, the largest single package was 40 pounds of diborane in a DOT-3AA2400 cylinder surrounded by dry ice in an insulated box. Gross shipping weight was 1300 pounds and Special Permit 970 required delivery within ten days.

Prospect of increased future demand for diborane rendered that package inadequate for the projected quantities and impractical from the standpoint of handling labor.

In accordance with the anticipated increased use of diborane, NASA awarded Contract Number NASW 1827 to Callery Chemical Company for the design and development of a container for shipment of approximately 200 pounds of liquid diborane.

This report, published in three volumes, describes the successful development of such a container. Volume I is the basic report, complete in itself; while Volume II (Appendix A) contains documents of record in correspondence with DOT to obtain a shipping permit, and Volume III (Appendix B) is the manual of Operating Instructions for use of the container.

SUMMARY

A container for shipping 200 pounds of diborane was designed, built, and tested. The Department of Transportation granted Special Permit No. 6522 for use of the container.

Work began 7 November 1968 on the program to design and develop an engineering prototype shipping container for diborane. Detailed specifications were prepared and submitted to four candidate sub-contractors for bids on container design and fabrication. Two bids were received, and the decision was made to award this sub-contract to CVI Corporation. Award of the sub-contract was postponed to consider the possibility of using the diborane container design for oxygen difluoride, but it was decided to proceed with design exclusively for diborane. The subcontract for container design and fabrication was approved by the Contracting Officer and signed by CVI Corporation on 20 February 1969.

The detail design was prepared by CVI with advice from Callery on the diborane system. Manufacturing drawings for approval were received from CVI on 17 April 1969, and were forwarded for NASA approval. The CVI design, with some modifications, was approved by JPL and Callery on 26 May 1969. Revised drawings were released for fabrication on 30 June 1969. Callery met with the Bureau of Explosives; and as a result, some changes were made for better protection against severe mishandling. After further discussions with JPL, Department of Transportation, and Bureau of Explosives the design was finalized.

Upon completing fabrication of components, the container assembly was started on 22 September 1969, at which time a Callery visit was made to CVI Corporation. Assembly and testing at CVI were completed on 8 December 1969, at which time Callery visited CVI for final inspection. The container was received at Callery on 17 December 1969.

Preliminary checkout of the container and facility were completed, and on 2 February 1970 diborane was first charged to the container. A small diborane leak was detected, and loading was discontinued after 25 pounds had been charged. Leakage which had developed in the level probe assembly was stopped on 13 February 1970, and subsequent low temperature tests confirmed that the entire system was tight.

The container was charged with 200 pounds of diborane and 100 pounds of dry ice; and the first storage test was started

on 6 April 1970. Container performance was disappointing; as dry ice lasted less than eight days, and diborane temperature reached the specified -35°C. during the tenth day. Diborane was unloaded from the container, and rate was shown to be well above the required 0.2 pound per second.

Heat transfer analysis following the unsuccessful storage test indicated the need for certain revisions to the container. After discussions with NASA and CVI, Callery wrote specifications for the necessary modifications; and the container was shipped from Callery on 11 August 1970. Agreement was reached between Callery and CVI, who then proceeded with container modifications and testing in their shop.

Vacuum retention testing of the modified container was successful; however, leakage into the insulation space developed at low temperatures encountered in thermal testing. The leak was located and sealed; after which the inner tank was heated to enable an effective pumpdown of the insulation space. Thermal tests at CVI using liquid nitrogen showed a significant reduction in the heat leak rate. The container was delivered to Callery 21 January 1971.

After cleanout with methanol, the container was dried in preparation for charging diborane. Heating the inner tank to assist drying caused a sharp rise in the insulation space pressure. Subsequent cooldown failed to restore the initial low pressure. Pressure rise in the insulation space was caused by non-condensables; so a system was setup to repump a better vacuum, achieving 15 microns at room temperature.

The container was precooled with liquid nitrogen, then charged with 200 pounds of diborane. The repeat storage test began on 22 April 1971. This successful storage test was terminated after 29 days. Time to -35°C. was over twenty days, and conservative extrapolation gives over 33 days to 0°C. The 0°C. maximum temperature is a practical limit corresponding to pressure of 400 psig; whereas the true limit is the maximum working pressure of 500 psig. Container performance was probably adequate to obtain a 20-day Special Permit; however, we recommended a 15-day permit to expedite delivery. A written presentation of the test data and drawings was prepared and submitted to Bureau of Explosives and Department of Transportation on 18 June 1971, along with application for a 15-day special permit.

All diborane was left in the container until 23 June 1971, at which time a sample was taken. After over 61 days there was negligible diborane decomposition. The container's center of gravity was experimentally determined while fully charged with diborane and dry ice. Diborane was then removed to the plant

storage system; and the container was cleaned and charged with methanol in preparation for the shipping test. DOT required that methanol shipment in this container be authorized by a special permit, included in the diborane permit.

DOT Office of Hazardous Materials drafted the special permit, which was then approved by the Federal Highway Administration. The Federal Railroad Administration, however, disclosed that DOT-OHM had added "Class A Poison" to the diborane classification. This prohibits rail express shipments, unless DOT were willing to make an exception to regulations; and could create other problems. DOT-OHM was notified to withhold issue of the special permit until the matter of classification had been resolved. Callery made a written presentation to DOT in support of our position that diborane does not qualify as a Class A Poison. Concurrently, requested information on safety relief devices was provided to Bureau of Explosives for their approval.

On 9 December 1971 DOT Office of Hazardous Materials ruled that "Class A Poison" was to be added to the "Flammable Compressed Gas" classification for shipment of diborane. We requested immediate issue of the special permit; and as a separate action, for DOT to consider waiver of the restriction against rail express shipment. DOT Special Permit Number 6522 was received 27 December 1971.

The shippping test with methanol was started on 12 January 1972 by motor freight shipment. The container was received in good condition at Callery's Lawrence, Kansas plant on 21 January 1972; and was shipped from Lawrence by motor freight on 28 January 1972. The shipping test was completed on 7 February 1972 when the container was received in good condition.

Several modifications to the permit were requested of DOT in March through May 1972, by letter and by visit to the DOT office. Permission for rail express shipment could not be granted with diborane classified as a Class A poison; however, variance for individual cases will be considered. Request for shipment of quantities less than 100 pounds was withdrawn, pending possible collection of additional data by NASA during use of the container. DOT issued First Revision to DOT Special Permit No. 6522 on April 1972, incorporating rewording recommended by Callery. Provisions to ship containers cooled or recooled to -70°C. (instead of -80°C.) were incorporated by Second Revision of the permit, issued 14 September 1972.

An operating manual with instructions for use of the container was prepared.

DESIGN AND CONSTRUCTION

SPECIFICATIONS

Detailed specifications were developed for submitting to subcontractors to use in preparing bids on container design and fabrication. These specifications, given in Table 1*, basically describe a 200 pound diborane shipping container designed for safety, reliability, durability, and convenience. Final specifications were to be developed in cooperation with the sub-contractor to be selected, with the above criteria as a guide to combine his knowledge of cryogenic containers with Callery's experience in diborane handling.

SELECTION OF SUB-CONTRACTOR

A survey was made to obtain a bidder's list of candidate sub-contractors for detail design and fabrication of the container. Although many companies are active in the field of cryogenic equipment, most of these were eliminated for one of the following reasons: (1) companies who only manufacture minor components, (2) companies too small and/or inexperienced to qualify, (3) companies who specialize in large equipment, and (4) companies who only offer standard containers and not custom designs. The list was thereby reduced to four companies:

1. CVI Corporation
   Columbus, Ohio

2. Chicago Bridge and Iron Company
   Pittsburgh, Pennsylvania

3. North American Phillips Company, Inc. (Cryogenic Div.)
   Ashton, Rhode Island

4. Ryan Industries (Div. of Cosmodyne Corporation)
   Louisville, Kentucky

A request for bid was submitted, with preliminary specifications, to all four companies. North American Phillips declined to bid on a custom-built container, and their standard containers did not meet the specifications. Chicago Bridge and Iron stated that their bids on small custom-manufacturing jobs would not be competitive with others in the field. Both CVI Corporation and Ryan Industries expressed a desire to bid, and representatives of both companies visited Callery to discuss

*All tables and figures are grouped at the end of each section in this report.

the specifications. These companies felt the specifications were reasonable and obtainable, and bids for container detail design and fabrication were received from both CVI Corporation and Ryan Industries. Several phone conversations were held with both while evaluating the bids.

A decision was made to award the sub-contract to CVI Corporation. A summary of the CVI proposal is given in Table 2; the complete proposal was quite detailed, including heat transfer calculations and preliminary container drawings (Figure 1 and Figure 2). In contrast, the bid from Ryan Industries gave very little information on design approach or other details; as the bid merely stated that they would meet the specifications. There was no factor in which the Ryan bid was superior to CVI; in addition, the Ryan bid was $2000 higher than the CVI bid.

The CVI Corporation sub-contract for container design and fabrication was approved by the Contracting Officer on 17 February 1969. This subcontract was fully executed with an effective date of 20 February 1969.

OTHER USES FOR CONTAINER

In a telephone conversation with NASA on 2 January 1969 two new uses for the diborane shipping container were discussed, as follows:

1. Use Container as Feed Tank

   It was desired to use the shipping container as a feed tank, which required an unloading rate of one pound per second with 100 psi chamber pressure and about 100 psi (maximum 200 psi) line pressure drop. In the absence of this information, bids for container design had been based on an unloading time of two hours. This new requirement was to be incorporated during the detail design of the container.

2. Use Container for Oxygen Difluoride

   It was desired to use the diborane shipping container design for oxygen difluoride (OF_2) shipment. Although there is some overlap in the liquid ranges of diborane and OF_2, there is a very significant difference in vapor pressures as shown in Figure 3. As a result a high pressure vessel would be required to ship OF_2 with dry ice refrigerant, or conversely an alternate refrigerant system such as liquid nitrogen would be required to ship OF_2 at lower temperatures. We encouraged the use of dry ice because

of the inherent simplicity and reliability of such a system. Use of dry ice eliminates the need for flow controls, the potential for overcooling of the diborane and other disadvantages of a liquid nitrogen system. In addition, the density of OF_2 is significantly higher than that of diborane; which would increase strength requirements, thereby increasing heat leak and weight.

The advantages and disadvantages of a container for use with both OF_2 and diborane were discussed in a meeting (attended by CVI Corporation representatives) on 21 January 1969. The dual purpose shipping container concept was judged impractical from technical and economic standpoints. Directions were given to proceed with the design of a container to be used exclusively for shipping diborane, in accordance with the original work statement.

CONTAINER DESIGN

The CVI design approach summarized in Table 2 incorporated the features of safety, reliability, and durability which we encouraged in the specifications. The container relies primarily on good insulation to achieve the required low temperature storage time. The design for dry ice addition not only helps meet that requirement, but also enables continued low temperature storage by recharging the dry ice.

NASA Recommendations

In accordance with NASA recommendations, the following modifications were incorporated in the design:

1. Use dip-tube outlet from the top rather than bottom outlet.

2. Dip-tube and valves to be 1/2-inch size or equivalent.

3. Valves to terminate in a replaceable "tee" fitting, so that the external lines can be purged.

4. Pressure rating of the tank to be increased to 500 psi.

5. The base on which the tank is mounted to extend a few inches beyond the envelope of the tank and valves.

6. Other types of level control devices to be investigated. It may be possible to make a multipoint level system using spring balanced floats and magnetic switches. Other than this, a bubble tube and differential pressure cell could be considered.

Unloading Rate

It became necessary to reduce the maximum unloading rate of diborane from the container, as it was not practical to design for one pound liquid diborane per second with 100 psi pressure drop. Based on 1/2 inch tubing, pressure drop for the tubing (20 feet equivalent length) would have been 200 psi at one pound per second; in addition, pressure drop for the valve would have been 400 psi. It was agreed that the maximum unloading rate should be reduced to 0.2 pound per second with 100 psi pressure drop.

Design Details

The diborane shipping container design details remaining were discussed in a meeting with CVI Corporation on 19 March 1969. Minor component and instrument specifications were completed, in addition to reviewing the general arrangement and structural design. Final container drawings for approval were prepared by CVI; these were received at Callery on 17 April 1969, and copies were forwarded to JPL for NASA approval. A number of discussions were held with CVI and JPL to resolve specifications for level instrumentation, materials of construction, and some minor component specifications and design details.

Liquid Level Instrument:

The liquid level instrument is a Robertshaw capacitance type with remote mounted transmitter. The level probe connects to the top of the sphere by a threaded joint. CVI Corporation recommended an epoxy adhesive for sealing the threaded connection. This epoxy (Armstrong Products Company, Inc. A-271 two-part adhesive) has good low temperature properties, but there was some question about its compatibility with diborane; so a test was setup at Callery to evaluate this thread sealant for diborane service. This test did not reveal any incompatibility; however, subsequent checking on the epoxy material showed that it contains certain chemical groups which could be reactive with diborane. As a result, Teflon tape was used in place of the epoxy material. A flanged connection had also been considered until it was learned that this still did not eliminate a threaded connection, as the probe would be threaded into the flange.

Stainless Steel Specifications:

Materials of construction for the diborane system were initially specified to be stainless steel, the type to be determined by fabrication requirements. This was based on some fifteen years of service for stainless steels 304 and 316 in Callery's low temperature diborane storage system and for carbon steel in the diborane production facility. The CVI design specified type 304 stainless steel for most of the diborane system. This specification was subsequently questioned by JPL, with the suggestion that all parts to be welded should be fabricated from 304L low carbon stainless steel or other stabilized low carbon type. It is correct that this would be better for welding if there is any corrosion, as the 304 stainless steel is susceptible to carbide precipitation at the grain boundaries around welds. CVI was given a list of six acceptable stainless steel types: 304L, 304ELC, 316L, 316ELC, 321, and 347.

Other Design Details:

Other minor changes were made in the design. Additional supports were provided at the base of the container to provide stability and safety in fork truck handling. The thermal plug for the dry ice chamber was to be sealed after insulating to maintain the low thermal conductivity of the polyurethane foam insulation. Additional items such as pressure gauge and valve specifications, cover fasteners for access to valves for loading and unloading, welding details, etc. were reviewed by Callery and necessary changes noted on drawings returned to CVI.

Design Approval & Modifications

The remaining design details were resolved between CVI, JPL, and Callery. Callery received NASA (JPL) approval of the design by telephone on 15 May 1969. On that date Callery authorized CVI Corporation to proceed with procurement of long delivery items. Upon receipt of the JPL confirming letter (dated 19 May 1969) on 26 May 1969, Callery sent a confirming letter to CVI. Callery's letter contained a list of all necessary modifications to the drawings, and one set of marked drawings was enclosed. Some of the more important modifications were as follows:

1. Inner vessel to be fabricated from type 304L stainless steel; and all other welded components which diborane contacts to be fabricated from 304L, 304ELC, 316L, 316ELC, 321, or 347 stainless steel on a best effort basis. Any items which could not reasonably be obtained in one of these six materials was to be fabricated from type 304

stainless steel on approval from Callery based on reported price and/or delivery time justification from CVI. The responsibility for meeting design requirements with these materials remained with CVI.

2. Use Teflon tape in place of the epoxy sealant for level probe and temperature probe connections, which are to be threaded connections as shown on the drawings.

3. Additional supports to be provided at the base of the container to provide stability and safety in fork truck handling. The container to be lifted by these horizontal structural member such that the forks do not contact the bottom head of the outer container.

4. The thermal plug to be sealed across the bottom after insulation by welding on a 0.031 inch thick cover.

5. The Whitey valves for pressure gauge shutoff and for gauge line purge are to be replaced by a more reliable type. Even a small amount of diborane decomposition could produce solids which may cause wear of the solid Teflon cylinder packing around the stem.

6. Change 1/4 turn fasteners to use No. 5 oval head stud instead of No. 5 wing head stud, at least on the removable side panel where susceptibility to damage is the greatest.

7. One additional drawing should be prepared to emphasize operating features of the container. Fabrication details should be deleted, and labeling should be by function rather than specification. All items routinely used by Callery or by the Customer when the container is in service should be shown on the drawing.

8. Provide stenciled and/or printed labels on the container to identify all valves, instruments, etc. to assure efficient and safe use of the container. Labels shall also be used for brief notes on operating instructions or notes on precautionary measures.

9. The audible and visual alarms when activated shall continue for a time of at least 72 hours providing the battery is maintained in accordance with CVI instructions.

CVI Corporation modified the container design in accordance with Callery's instructions, and revised drawings were received by Callery on 7 July 1969. The following additional changes were made to those drawings.

1. Inner tank to be stamped as a coded vessel. Initially the vessel was to be built in accordance with the code, but not stamped; and this was acceptable to the Bureau of Explosives and Department of Transportation. Certain government agencies, however, require that all pressure vessels operated on their premises be stamped as coded vessels; therefore, it was felt that not coding the vessel would impair its usefulness.

2. The vent system was revised to eliminate a low point trap where liquid could have collected and been expelled into the vent line. This change was accomplished by removing the vent tubing inside the inner vessel, which still leaves adequate working volume. The volume below the vent line point of entry is over 100 gallons; whereas, even at 0°C., the volume occupied by 200 pounds of diborane is less than 85 gallons.

One design change was made as a result of a meeting with the Bureau of Explosives. At the top of the container, piping which is open to the inner tank was to be protected by one-eighth inch thick steel plate.

One additional functional assembly drawing was prepared to illustrate operating features of the container. The essential parts of that drawing are reproduced in Figure 4 and 5.

FABRICATION AND ASSEMBLY

Approximately five months were required from approval of drawings to delivery of the vessel to Callery. This was made possible by early approval of three long delivery items: (1) inner vessel heads, (2) inner vessel support rods, and (3) liquid level instrumentation.

CVI released the drawings for fabrication on 30 June 1969. Procurement was initiated immediately by CVI on all of the materials required for the diborane shipping container. In general there were few delays encountered. Procurement created no problems, even with the specification of 304L or equivalent for diborane-contact parts to be welded. The inner tank heads were shipped on 4 August 1969.

Close contact with CVI was maintained during this phase of the program. The inner vessel fabrication and tests were completed; and successful results on weld radiographing tests were confirmed on 3 September 1969. A minor delay occurred when one of CVI's suppliers encountered difficulty rolling the 2-1/2 X 2-1/2 inch angle iron reinforcing members. After several tries the supplier finally did a job which was judged unacceptable, and this procurement was changed to another source. No difficulty was encountered with the new supplier, and the angle iron

reinforcing members for the inner vessel were received, fit up, and welded on. The inner vessel hydrostatic test was completed on 16 September 1969, in the presence of the code inspector. After a minor delay to obtain the correct nuts for the inner vessel support rods, the main assembly was started on 22 September 1969. A Callery visit was made to CVI when the inner vessel was positioned and secured in the outer shell. Callery was favorably impressed with the work performed by CVI.

Details of the protective plates recommended by Bureau of Explosives were worked out with CVI, and installation was completed. Rework on the sheet metal at the top became necessary when some of the metal work as originally installed did not meet CVI standard, so the affected sections were replaced. Since this required welding to the outer shell, the final leak test was delayed until that welding was completed.

CONTAINER TESTING AT CVI

In addition to the inner vessel weld radiograph and hydrostatic tests mentioned above, the inner tank and associated piping passed the helium mass spectrometer leak test. Also the outer vessel (annular space between inner tank and outer shell) successfully passed helium mass spectrometer leak tests initially, after welding on the protective plates, and after welding on new sheet metal around the top enclosure.

The vacuum retention test was performed after charging the perlite and evacuation of the annular space. This evacuation required continual pumping from 13 November 1969 to 4 December 1969 because of moisture in the perlite. Even with heat on the outer shell and repeated pressurization-pumpdown cycles, this evacuation required three weeks to complete. After final pumpdown there was no ice in the cold trap, and pressure was down to 7.5 microns at the time of seal-off. Following immediately, the vacuum retention test demonstrated that the annular space was adequately sealed. As shown in Figure 6, pressure reached equilibrium at 22 microns (at 74°F.) two days later; and this remained the same for the duration of the test, concluded on 8 December 1969. The final step was leak testing of the diborane system after cooling with dry ice.

A Callery visit was made to CVI on 8 December 1969 to witness these final tests and to inspect the container prior to shipment. Accessible parts of the container, such as external piping and instruments, were found to be satisfactory. Instructions were provided for component labeling prior to delivery. Production orders, inspection record sheets, and test data were reviewed; and everything was found to be in order.

CONTAINER DELIVERY

The container was shipped from Columbus, Ohio by CVI on 9 December 1969 and received by Callery on 17 December 1969. The container was crated for this shipment to preserve appearance and it was received with no apparent damage. The container is identified by National Board number 19 and CVI Serial Number 231. The inner vessel is coded for a maximum working pressure of 500 psig at -320 to +100°F.

MODIFICATIONS

Following Performance Test # 1 modifications were made to the container to improve its thermal efficiency. Primarily heat leak was reduced by redesign of the neck area and restoring the low pressure in the insulation space.

The bellows protective sleeve, initially fabricated from stainless steel, was replaced with a sleeve of Teflon 0.031 inch thick.

The side walls of the thermal plug, initially fabricated from stainless steel, were also replaced with 0.031 inch thick Teflon.

The vacuum filter assembly was shortened to a length of 27 inches to eliminate possibility of a thermal short between the inner and outer vessels.

Pressure in the insulation space was reduced to less than ten microns (Figure 12), and a bellows seal high vacuum valve was added to facilitate any future need to repump (Figure 13).

These modifications were incorporated in the as-built drawings.

DRAWINGS

Following is a complete list of as-built drawings for fabrication and assembly of the 200-pound diborane shipping container. Original tracings and copies of these drawings have been supplied to NASA separate from this report, in accordance with the terms of the contract.

A458-5800	Flow Schematic
A458-5801	Final Assembly
A458-5802 Sh 1 & 2	Piping and Instruments
A458-5803 Sh 1 & 2	Main Assembly
A458-5804 Sh 1 & 2	Inner Vessel - Assembly Of
A458-5805	Inner Vessel - Details
A458-5806 Sh 1 & 2	Top Head - Assembly Of
A458-5807	Top Head - Details
A458-5808	Vacuum Filter - Assembly Of
A458-5809	Bottom Head - Assembly & Detail
A458-5810	Thermal Plug - Assembly Of
A458-5811	Suspension Bracket - Assembly Of
A458-5812 Sh 1 & 2	Suspension Bracket - Details
A458-5813	Bracket-Pressure Gage
A458-5814	Standoff Ring - Assembly Of
A458-5815	Lifting Bracket Details
A458-5816	Outer Shell - Assembly Of
A458-5817	Top Panel - Assembly Of
A458-5818 Sh 1 & 2	Side Panel - Assembly Of
A458-5819	Valve Cover - Assembly Of
A458-5820	Fill Port - Perlite, Assembly & Detail
A458-5821 Sh 1 & 2	Electrical Schematic & Panel Layout

TABLE 1

SPECIFICATIONS FOR DIBORANE SHIPPING CONTAINER

General

These specifications define a refrigerated, insulated, portable container to ship liquid diborane. The container shall be designed for safety, reliability, and durability.

The container, including all its component parts and auxiliaries, shall meet all applicable codes and regulations.

All parts which contact diborane shall meet material and design specifications prescribed by Callery Chemical Company.

Capacity

The container shall be designed to ship 200 pounds net weight of liquid diborane under the conditions stated below.

Shipping Temperature

Temperature of liquid diborane, initially at -80°C., shall not exceed -35°C. after thirty days with an ambient temperature of 25°C.

Diborane Container Volume

If the temperature rises to -35°C., the diborane expansion in an additional five days time shall not result in a liquid volume greater than the design working volume of the vessel; diborane temperature after the five days shall not be higher than 0°C.

Diborane Container Pressure

Design working pressure shall be not less than 25 percent in excess of the diborane vapor pressure at the maximum temperature (five days after -35°C.). The container shall also be designed for full vacuum service.

Refrigerant Requirements

A method shall be provided to recharge refrigerant for storage periods longer than thirty days and/or to maintain lower temperature (within the range of -35 to -80°C.). A commercially available refrigerant shall be used.

Table 1 - Specifications For Diborane Shipping Container (cont)

Container Configuration

In general the container shall be a low-profile design, the height of which shall not exceed 78 inches.

Container Handling

The container shall be skid mounted with slots for fork truck handling, and shall also be designed with lifting lugs for moving with a crane.

Container Durability

The container, including all its component parts and auxiliaries, shall be designed to withstand a force of at least three G's in all directions without causing any leakage or damage.

Process Piping

Piping shall include: (1) an internal dip tube to enable removal of the diborane as a liquid through a manual shutoff valve in a time not to exceed two hours and (2) a line connected to the container top for venting or pressurization through a manual valve.

All connections shall be welded or flanged. Flanges will be of a type suitable for cryogenic service. The entire system shall be free of leaks at the maximum working pressure of the vessel. Valves shall be accessible, but protected from damage.

Safety Relief

A rupture disk shall be connected directly to the container top, with a relief valve located immediately downstream of the rupture disk and a pressure gauge between to indicate failure or leakage of the rupture disk. Pressure settings shall be determined by the design operating conditions, and shall be consistent with all applicable codes.

Instruments

The following instruments shall be included: (1) a pressure gauge with safety release back plate, to be connected to the container top through a manual shutoff valve and an excess flow valve: (2) a temperature indicator covering at least the range from -100 to +20°C.; in conjunction with or separate from the indicator, there shall be an audible alarm activated by temperature higher than -35°C.; and (3) your recommendations for a suitable level indicator are solicited.

Table 1 - Specifications For Diborane Shipping Container (concluded)

Indicators shall be mounted to be visible from the side, but positioned to prevent damage under the forces stated above.

Solvent Resistance

All diborane contact parts shall also be selected for resistance to any undesirable effects due to contact with naphtha, isopropyl alcohol, or methyl alcohol.

Note: A copy of the manual containing diborane properties and handling information was included with the specifications in requesting bids for container design.

TABLE 2

SUMMARY OF CVI CORPORATION PROPOSAL
FOR
DIBORANE SHIPPING CONTAINER DESIGN AND FABRICATION

1. Scope and Price

Design, fabricate, deliver, and furnish field service for a prototype diborane shipping container for a total fixed price of $23,000 f.o.b. Callery Chemical Company.

2. Schedule

a. Complete design and drawings six weeks after receipt of order.
b. Complete fabrication four months after approval of design drawings.
c. Complete field service for vessel testing four to six weeks after delivery of the vessel.

3. Design Approach

a. Refrigeration of the diborane during shipment by reflux at a dry ice cooled top part of the container wall.
b. Evacuated powder type insulation, requiring only moderate vacuum level.
c. See Figure 1 and Figure 2.

4. Specifications

a. Capacity for 200 pounds of diborane at 0°C. with 10% ullage.
b. Time for heatup from -80°C. to -35°C. to be not less than thirty days. Time for heatup from -35°C. to 0°C. to be about twenty days.
c. Inner shell to be 36 inch diameter sphere, 5/16 inch thick; to comply with ASME Code for 400 psi internal pressure.
d. Outer shell to be a vertical cylinder. All lines, valves, gauges, etc. to be within a protected envelope not to exceed 6'-6" height. Provisions will be made for handling by crane or by fork lift truck.
e. Design for 8g loading vertically down and in all horizontal directions, and 4g loading vertically up.

Table 2 - Summary of CVI Corporation Proposal (Cont)

5. Instruments

The design shall include a temperature indicator, a pressure indicator, and level indicator. Alarms will be included to be activated by temperature above -35°C. or pressure above 180 psi. The inner vessel will be protected by a rupture disk and relief valve.

6. Guarantee

The diborane vessel will be guaranteed for a period of one year against failure due to defects in material or workmanship.

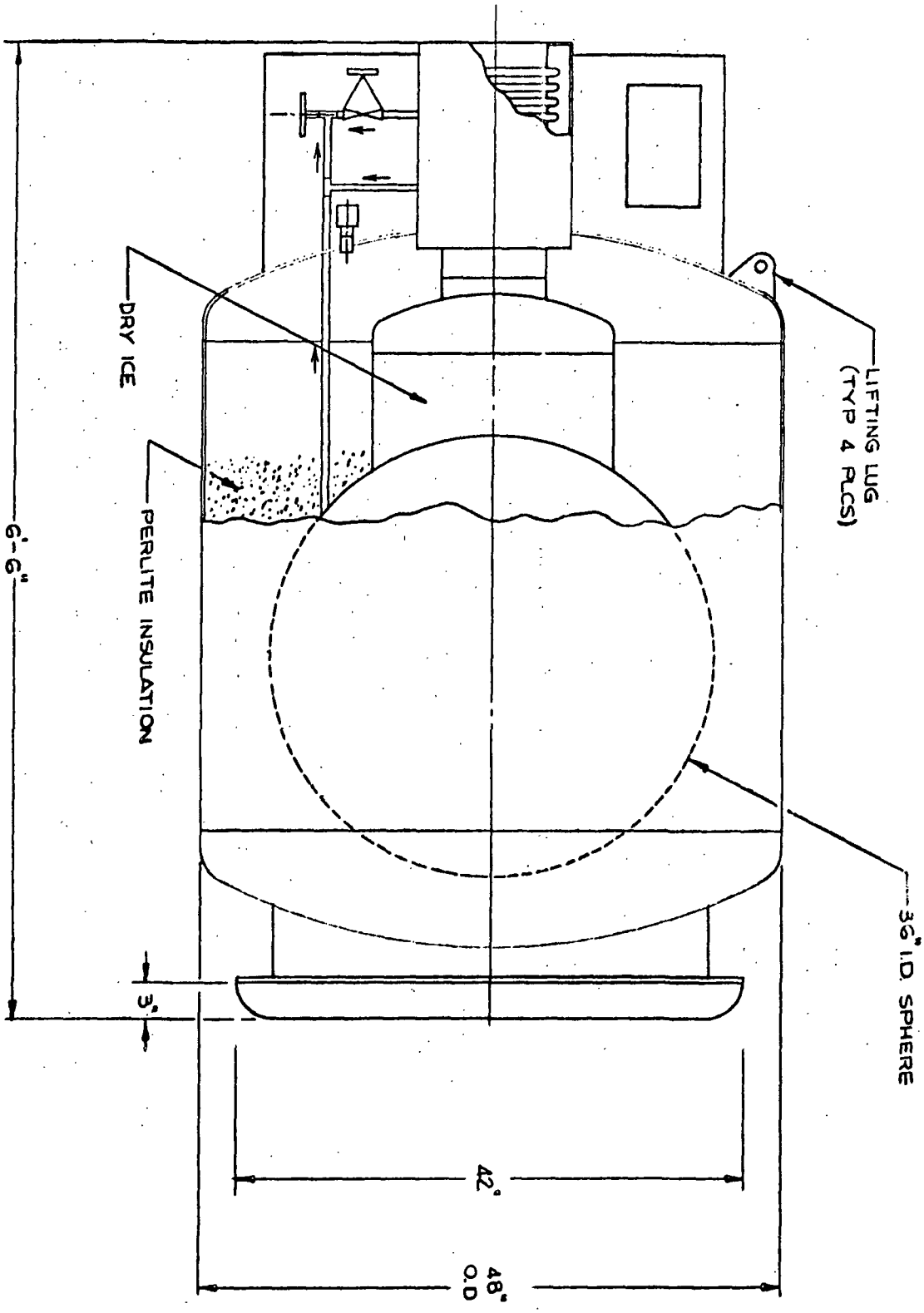


FIGURE 1

PRELIMINARY DIAGRAM OF
DIBORANE SHIPPING CONTAINER

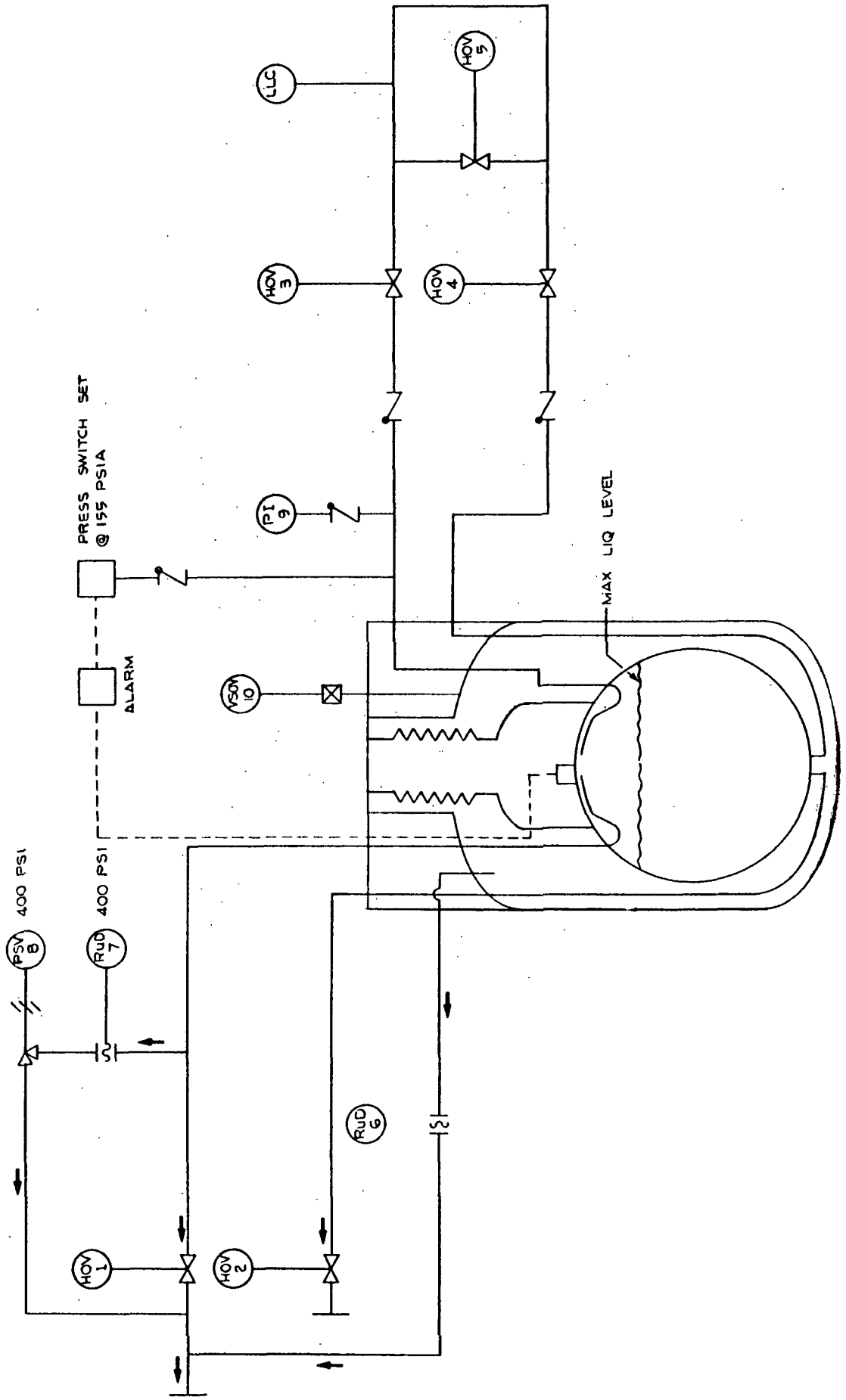


FIGURE 2

PRELIMINARY FLOW SCHEMATIC OF DIBORANE SHIPPING CONTAINER

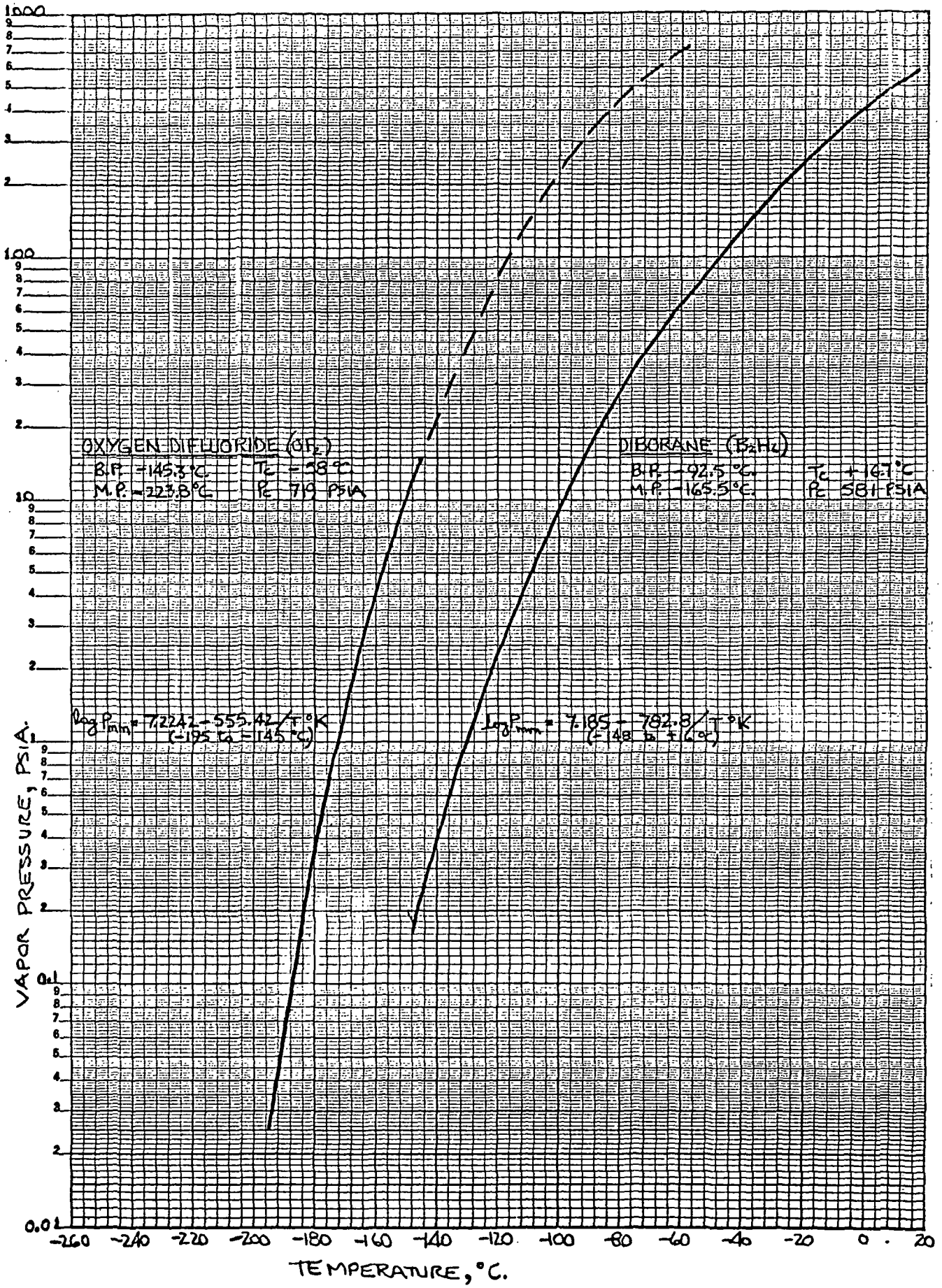


FIGURE 3

DIBORANE & OXYGEN DIFLUORIDE VAPOR PRESSURE

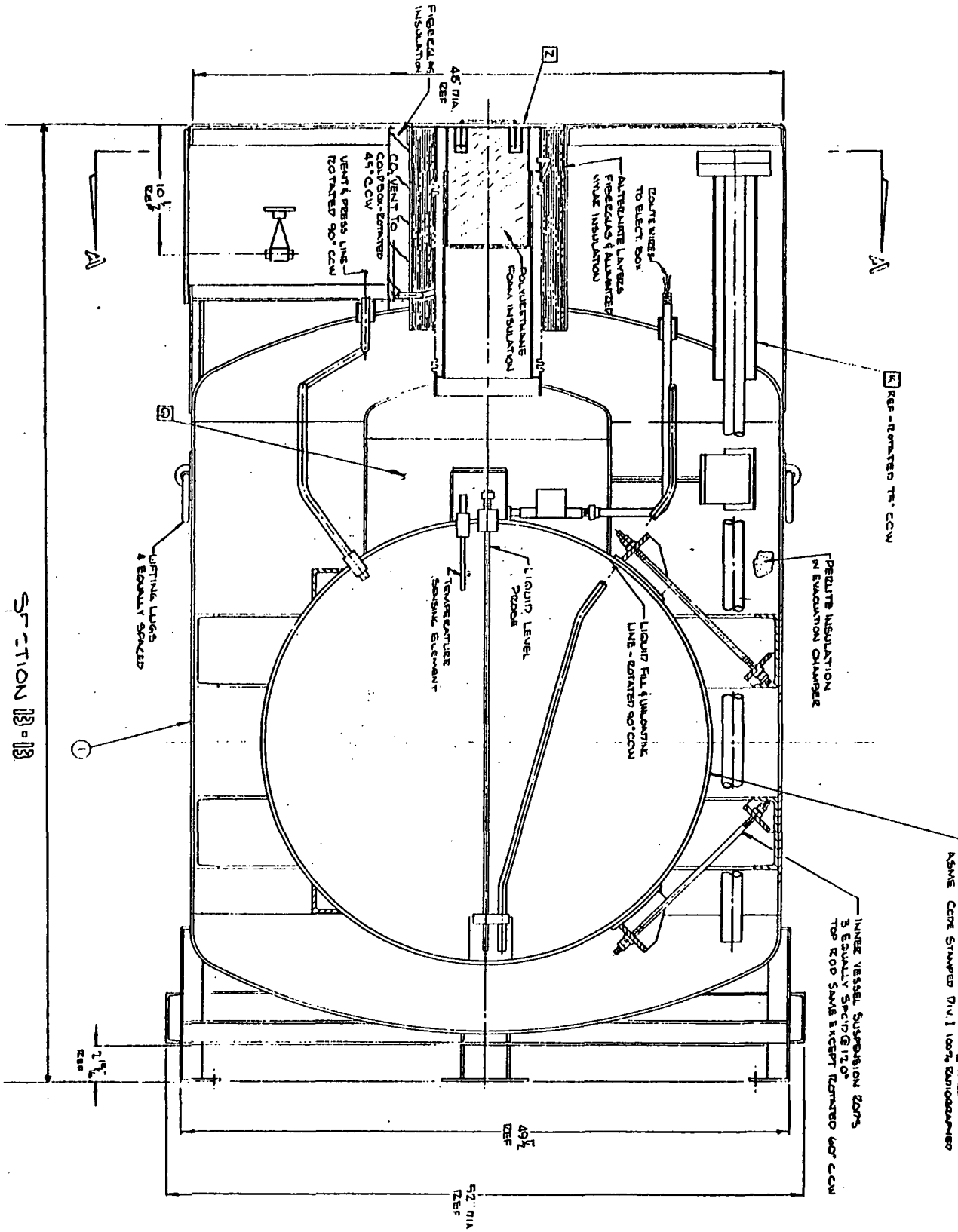
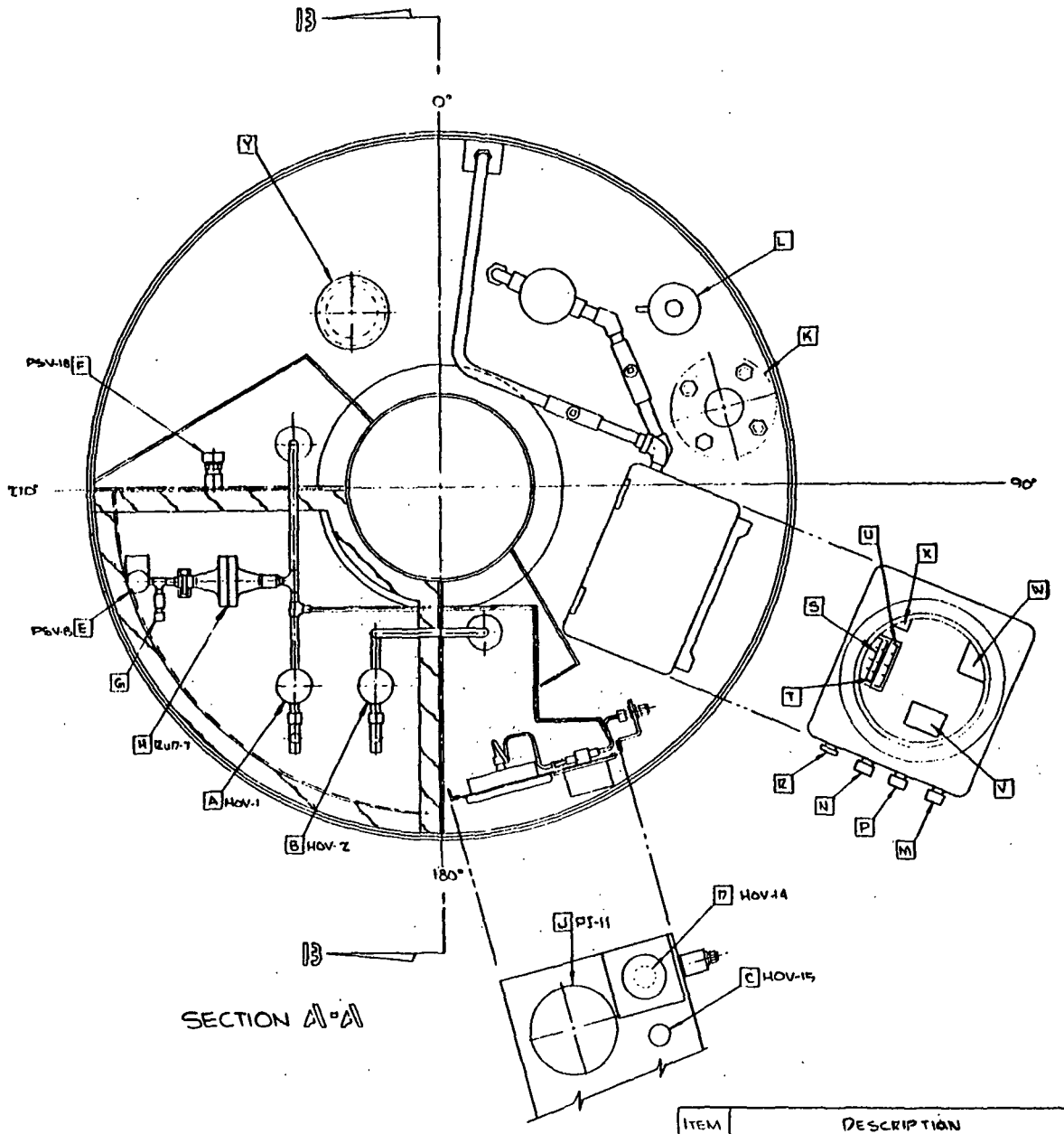


FIGURE 4

DIBORANE SHIPPING CONTAINER, ELEVATION VIEW



ITEM	DESCRIPTION
A	VENT VALVE & PRESSURIZATION VALVE
B	FILL VALVE & LIQUID UNLOADING VALVE
C	PRESSURE GAGE ISOLATION VALVE
D	PRESSURE GAGE PURGE VALVE
E	SAFETY VALVE SET @ 550 PSIG
F	SAFETY VALVE SET @ .5 PSI
G	EXCESS FLOW VALVE
H	RUPTURE DISC SET @ 550 PSIG
J	PRESS. GAGE -30" VAC -0 TO 1000 PSIG RANGE
K	FILTER ASSY W/HASTINGS DV6M VAC GAGE
L	LINDE OPERATOR F/2" PUMP OUT VALVE
M	TEST-PUSH BUTTON
N	SILENCE-PUSH BUTTON
P	LEVEL INDICATOR PUSH BUTTON
Q	DRY ICE CHAMBER
R	ON-OFF SELECTOR SWITCH
S	VOLTMETER-LINEAR SCALE
T	LIQUID LEVEL INDICATOR
U	TEMPERATURE INDICATOR (-130° C. TO +130° C.)
V	CONTROL RELAY - 24V
W	BATTERY - 27V, 3,400 MAH
X	VISUAL & AUDIO ALARM
Y	PERLITE FILL PORT
Z	DRY ICE CHAMBER FILL & VENT PORT

FIGURE 5

DIBORANE SHIPPING CONTAINER
PLAN VIEW

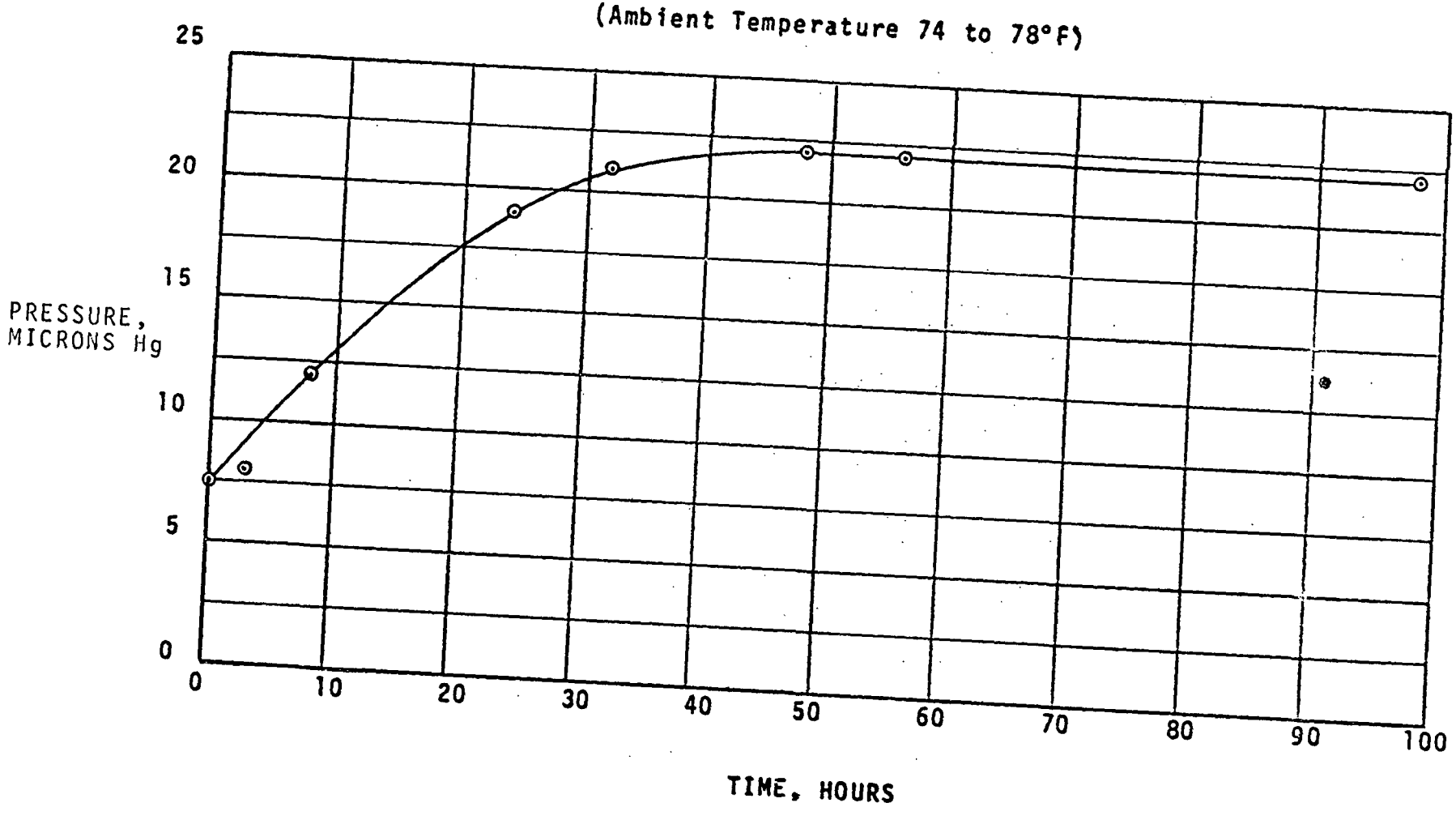


FIGURE 6

VACUUM RETENTION TEST @ CVI (DECEMBER 1969)
DIBORANE SHIPPING CONTAINER ANNULAR SPACE

PERFORMANCE TEST #1

LOADING FACILITY

A test site was prepared for filling, storing, and unloading the container. The shelter was completed, and piping was installed to this area. Provisions were made to withdraw diborane from the Callery storage tank and condense it into the container on a scale to calibrate the level instrument. Further provisions were made to unload the container by nitrogen pressurization, removing liquid diborane from the container through the dip tube. Other piping provided the necessary purge, vacuum, and venting capabilities. A diagram of the piping is shown in Figure 7.

The Callery test facility was prepared prior to delivery of the container. Upon receipt of the container, its external piping was connected to the loading, unloading, and purge lines of the test facility.

CHECKOUT AT CALLERY

Vacuum Retention

After receiving the container, periodic measurements of pressure on the annular (insulation) space were made. The results, plotted in Figure 8, show a slow rise in pressure (loss of vacuum). The rise from 45 microns to 180 microns was acceptable, as CVI felt their design would still be quite safe even at 200 microns. Based on their past experience CVI attributed the rise to movement of the perlite during shipment, because leakage invariably causes a much higher rate of pressure rise.

Unloading Rate with Methanol

The container was filled with methanol for cleanout, after first measuring tare weight (2615.5 pounds). Container volume was confirmed by weight of methanol required for filling. Methanol was pressurized from the container dip tube into the diborane piping for cleanout.

While unloading methanol from the container, flow rate measurements were made with various pressure drops. These data, listed in Table 3 and plotted in Figure 9, conform to the equation:

$$x = 9.52 \log y - 10.4$$

where x = flow rate, gallons per minute
y = pressure drop, psi

Table 3A gives a more general analysis of the methanol unloading test data, also shown in Figure 9A. Although these data were not in the Reynolds Number range of interest for diborane unloading, they did suggest that the required 0.2 pound per second diborane unloading rate could be achieved with little difficulty (see also "DIBORANE UNLOADING TESTS", pages 29 and 30).

Instruments and Controls

Preliminary checkout of instruments was completed. The level indicator had to be calibrated with diborane rather than methanol; so that was done when charging. The level instrument zero was preset by CVI and did not need to be changed, but the span was reset using the scale as a basis for diborane level. While transferring methanol, the scale was rechecked against other scales to confirm calibration. During methanol flow rate tests, the container pressure gauge was checked against other calibrated gauges. There was a small difference between temperature of methanol charged and the container temperature indicator reading. It was discovered that low ambient temperature had a noticeable effect on battery output voltage. The battery manufacturer stated that even at -20°F. there should still be reasonable output at somewhat reduced voltage. The alarm would still operate properly, but not as loud or as long.

Drying of the container by nitrogen purging and vacuum was completed on 28 January 1970. The facility installation and leak test were completed by that time, and preparations were made to begin filling the container. Diborane production was scheduled to have the required amount in storage at the desired time.

DIBORANE CHARGE

Charging of diborane into the container was started on 2 February 1970. After starting late in the day, approximately 25 pounds of diborane was loaded in 30 minutes. As expected, pressure rose in the container because of diborane vaporization while the inner tank was being cooled. A slight diborane odor was detected during filling operations, but no leaks were discovered by test of external piping. Overnight the diborane odor became more pronounced, and its source was traced to the dry ice chamber. This suggested either the level probe or temperature probe as source of leakage.

On 2 February 1970 the diborane was unloaded back into the storage system, after which the container was purged with nitrogen to remove practically all of the diborane to make the container safe for maintenance. After removing the residual dry ice, the cover was removed from theprotective enclosure around

the top of the temperature and level probes. No leakage was detected at 50 psig, but leakage occurred in the level probe assembly when the pressure was raised to 75 psig.

Purging of the container was continued to assure safety during maintenance work. The necessary special tools were fabricated by CVI Corporation, and on 13 February 1970 a CVI representative visited Callery to assist in making repairs. As expected, the temperature probe was tight; and the level probe assembly was slightly loose. Both probes appeared to be well sealed to the container, as only the Teflon seal within the level probe assembly had relaxed after cooling. After tightening, no leakage could be detected. The container was then cooled and tested several times with no evidence of leakage. The level probe manufacturer reported that they would not anticipate leakage after following this procedure.

Diborane was charged into the container on March 9, 10, 25, and 26; and the last of the 200 pounds was charged on 6 April 1970. Container loading proceeded without difficulty. No leakage of diborane could be detected after the earlier repair and test. Delays were caused by weather, scheduling, and other factors unrelated to container or facility performance. The level indicator was reset when the container was about half full.

THERMAL TEST # 1

Immediately after the container was fully charged with diborane on 6 April 1970, the dry ice chamber was packed full of dry ice. Net weight of ice was 100 pounds, bringing the gross shipping weight up to 2913 pounds. The container was sealed as it would be for shipment, and ambient temperature was set to control 75 to 80°F. during the test. Temperature, pressure, and weight were recorded twice a day; other readings which required removal of the side panel, were recorded less frequently.

Test data are listed in Table 4 and plotted in Figure 10. Container performance was completely unsatisfactory, as all of the dry ice was gone in 7-1/2 days and diborane temperature rose to -35°C. in 9-1/2 days. Design specifications were for a time of 30 days before temperature reached -35°C.

The time to reach -35°C. might have been extended by as much as five days if initial diborane temperature had been at -78.5°C. (dry ice temperature). The container temperature indicator read the initial diborane temperature as -70°C.; however, it now appears that the thermocouple was influenced by the dry ice. Actual initial diborane temperature, based on vapor pressure data, is believed to have been about -54°C.

The test was continued until near the end of the eleventh day, at which time more dry ice was added. Thereafter dry ice was recharged as often as necessary, while additional data were recorded to obtain further information about heat leak rate. Data are shown in Figure 11.

TEST DATA ANALYSIS

Test data were reported promptly to CVI Corporation, the sub-contractor who designed the container for 30-day storage time. At the same time Callery personnel proceeded with independent analysis of the data in an effort to interpret results. The conclusion reached from the Callery analysis was that excessive heat leakage occurred for two reasons: (1) pressure rise in the insulation space resulted in a substantial increase in thermal conductivity through the perlite insulation and (2) design in the neck area permitted a very significant heat leak, some of which occurred through direct metal connections between the dry ice chamber and the surroundings.

Based on Callery's heat transfer anlaysis, the following recommendations were made to CVI Corporation:

1. Repump insulation space to below 50 microns and maintain no higher than 100 microns.

2. Fabricate a new thermal plug with stainless steel retainer replaced by material with low thermal conductivity.

3. Remove the stainless steel sleeve which protects the bellows and replace it with a material having low thermal conductivity.

CVI agreed with these recommendations, and added the possibility of the vacuum pump-out filter housing causing a thermal short between the inner and outer vessel. Arrangements were made to proceed with the necessary modifications.

DIBORANE UNLOADING TESTS

Diborane was unloaded from the container with no problems, and rate was higher than the minimum requirements. Specification for unloading rate was at least 0.2 pound liquid diborane per second with pressure differential of 100 psi. Actual unloading rate of over 0.2 pound per second was obtained with a pressure differential of only 20 psi.

Unloading rate was determined by measuring the time required for a given change in container gross weight, with regulated pressurization of the container and back pressure control on liquid from the dip tube. Data measurements with liquid diborane temperature of -38°C. were as follows:

Pressure, Psig		ΔP	Flow Rate	
Container	Outlet	PSI	Lb/Sec.	GPM
250	230	20	0.23	4.6
265	230	35	0.30	6.0

Unloading would normally occur at a temperature lower than the -38°C. used for testing; therefore, the higher density at lower temperature would permit still higher rates. Even at -38°C. the unloading rate should be over 0.4 pound per second with 100 psi pressure differential. Analysis of diborane unloading data is included in Table 3A and in Figure 9A, along with the methanol unloading data. Reynolds Number corresponding to methanol data is too low to be of interest for unloading diborane. Relating the methanol data to diborane could be misleading, because diborane tends to behave as a somewhat compressible liquid.

Following unloading tests, the container was cleaned by the procedure Callery had previously developed for cleaning diborane cylinders:

1. Purge with nitrogen.

2. Wash with naphtha.

3. Wash with isopropyl alcohol.

4. Wash with methyl alcohol.

After completion of these steps, the container was washed with water in anticipation of return to CVI for revisions.

TABLE 3

UNLOADING TEST DATA
OBTAINED WITH METHANOL (-1°C.)*

Container Pressure, psig	Downstream Pressure, psig	Pressure Differential, psi	Methanol Weight lbs.	Flow Time, min.	Flow Rate, lb/sec.	Flow Rate, gal/min
280	250	30	50	1.998	0.42	3.71
290	250	40	50	1.555	0.54	4.76
200	150	50	50	1.268	0.66	5.84
200	150	50	50	1.285	0.65	5.76
250	150	100	50	0.875	0.95	8.47
200	50	150	50	0.725	1.15	10.2
200	50	150	50	0.732	1.14	10.1
200	100	100	50	0.839	0.99	8.83
200	100	100	50	0.848	0.98	8.74
200	150	50	50	1.280	0.65	5.79
200	150	50	50	1.275	0.65	5.81

$x = 9.52 \log y - 10.4$

where x = flow rate, gallons per minute
y = pressure differential, psi

Smoothed Data:

5.76 GPM @ 50 psi
8.63 GPM @ 100 psi
10.3 GPM @ 150 psi

*Density 50.6 lbs/ft.^{3}
Viscosity 0.8 cp.

TABLE 3A

ANALYSIS OF
UNLOADING TEST DATA

$$\rho \Delta P / \dot{m}^2 = K / 2 g_c A^2$$

where $K = F(Re)$; $Re = \dot{m}/\mu(4/\pi d)$

$\rho = 50.6$ lb/ft^3 for Methanol @ -1°C
$\rho = 21.6$ lb/ft^3 for Diborane @ -38°C

$\mu = 5.38 \times 10^{-4}$ lb/ft sec for Methanol @ -1°C
$\mu = 0.477 \times 10^{-4}$ lb/ft sec for Diborane @ -38°C

$g_c = 32.2$ lb-m ft/lb-f sec^2
A = Area ft^2

ΔP = lb-f/in^2
$\dot{m}$ = lb-m/sec
} table, below

let K contain the conversion factor 144, to use ΔP as lb-f/in^2 instead of lb-f/ft^2.

	ΔP Pressure Differential psi	$\dot{m}$ Flow Rate lb-m/sec	[See Figure 9A] $\frac{\rho \Delta P}{\dot{m}^2}$	$\frac{\dot{m}}{\mu}$
METHANOL				
	30	0.417	8720	775
	40	0.536	7050	996
	50	0.657	5860	1220
	50	0.648	6020	1200
	100	0.952	5580	1770
	150	1.15	5750	2140
	150	1.14	5840	2120
	100	0.993	5130	1850
	100	9.983	5240	1830
	50	0.651	5970	1210
	50	0.654	5910	1220
DIBORANE				
	20	0.23	8170	4820
	35	0.30	8400	6290

TABLE 4

CONTAINER PERFORMANCE DATA - TEST # 1
200# DIBORANE SHIPPING CONTAINER

TIME	DATE	NUMBER OF DAYS	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F.	SCALE READING LBS.	DRY ICE LBS.	CONTAINER % LI*	CONTAINER % VI*	CONTAINER VACUUM MICRONS	REMARKS
1555	4/6/70	0	-69	65	55	2913	100	58	82	350	Start test
2000	4/6/70	0.17	-73	67	71	2910	97	-	-	-	
2355	4/6/70	0.33	-73	68	70	2907	94	-	-	-	
1000	4/7/70	0.75	-73	68	72	2901	88	-	-	-	
1615	4/7/70	1.01	-73	68	75	2897	84	-	-	-	
0025	4/8/70	1.35	-73	68	67	2892.5	79.5	-	-	-	
0830	4/8/70	1.69	-73	68	80	2887.5	74.5	-	-	-	
1000	4/8/70	1.75	-73	68	73	2887	74	-	-	-	
1550	4/8/70	2.00	-73	69	81	2883.5	70.5	-	-	-	
0945	4/9/70	2.74	-73	70	76	2873.5	60.5	-	-	-	
1035	4/9/70	2.78	-73	70	80	2873	60	-	-	-	
1245	4/9/70	2.87	-73	70	79	2871.5	58.5	58	82	375	Checked TI; -68°C
1535	4/9/70	2.99	-69	71	78	2870	57	-	-	-	
0815	4/10/70	3.68	-69	75	81	2861.5	48.5	-	-	-	
1010	4/10/70	3.76	-69	75	76	2860.5	47.5	-	-	-	
1500	4/10/70	3.96	-69	75	80	2858.5	45.5	-	-	-	
1945	4/10/70	4.16	-69	76	80	2855	42	-	-	-	
2400	4/10/70	4.34	-69	76	77	2853	40	-	-	-	
1050	4/11/70	4.79	-69	76	78	2846	33	-	-	-	
1800	4/11/70	5.09	-69	76	78	2842	29	-	-	-	
1340	4/12/70	5.91	-69	76	79	2830	17	-	-	-	
2335	4/12/70	6.32	-69	76	82	2824.5	11.5	-	-	-	
0805	4/13/70	6.67	-69	77	77	2820.5	7.5	-	-	-	
0845	4/13/70	6.70	-69	77	78	2820.5	7.5	-	-	-	
1535	4/13/70	6.99	-68	80	76	2817.0	4.0	-	-	-	
1640	4/13/70	7.03	-66	80	80	2816.5	3.5	-	-	-	
2200	4/13/70	7.25	-64	81	80	2815	2.0	-	-	-	
0835	4/14/70	7.69	-54	90	76	2813	0.0	-	-	-	
0855	4/14/70	7.71	-54	90	78	2813	-	-	-	-	
1200	4/14/70	7.84	-50	96	71	2813	-	-	-	-	
1510	4/14/70	7.97	-48	100	80	2813	-	-	-	-	
1700	4/14/70	8.04	-47	103	80	2813	-	-	-	-	

*LI = Level Indicator; VI = Voltage Indicator

TABLE 4 (Continued)

CONTAINER PERFORMANCE TEST DATA
200# DIBORANE SHIPPING CONTAINER

TIME	DATE	NUMBER OF DAYS	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F.	SCALE READING LBS.	DRY ICE LBS.	CONTAINER % LI	CONTAINER % VI	CONTAINER VACUUM MICRONS	REMARKS
1915	4/14/70	8.14	-46	107	75	2813	-	-	-	-	
2130	4/14/70	8.23	-45	109	73	2813	-	-	-	-	
2350	4/14/70	8.33	-43.5	111	78	2813	-	-	-	-	
0755	4/15/70	8.67	-40.5	121	72	2813	-	-	-	-	
0905	4/15/70	8.72	-40.5	121	78	2813	-	75	82	375	
1155	4/15/70	8.83	-40	126	79	2813	-	-	-	-	
1540	4/15/70	8.99	-39.5	130	76	2813	-	-	-	-	
1555	4/15/70	9.00	-38	131	77	2813	-	-	-	-	
1715	4/15/70	9.06	-37.5	135	75	2813	-	-	-	-	
2150	4/15/70	9.25	-37	138	83	2813	-	-	-	-	
2400	4/15/70	9.34	-36	140	76	2813	-	-	-	-	Alarm silent
0800	4/16/70	9.67	-33	150	77	2813	-	-	-	-	Alarm on
0840	4/16/70	9.70	-33	150	74	2813	-	-	-	-	
1150	4/16/70	9.83	-32	155	79	2813	-	78	82	375	
1530	4/16/70	9.98	-31	160	78	2813	-	-	-	-	
1915	4/16/70	10.14	-30	165	77	2813	-	-	-	-	
2350	4/16/70	10.33	-30	171	77	2813	-	-	-	-	
0755	4/17/70	10.67	-28	181	75	2813	-	-	-	-	
1006	4/17/70	10.76	-27.5	185	80	2813	-	78	83	380	Start to remove plug
1015	4/17/70	10.76	-26	185	-	-	-	-	-	-	Start to add dry ice
1021	4/17/70	10.77	-27	183	80	2909.5	96.5	-	-	-	Ice level below neck
1031	4/17/70	10.78	-	-	-	-	-	No alarm sounded			Ice addition finished
1033	4/17/70	10.78	-32	176	78	2919.5	106.5	0	83	380	Thermal plug in place

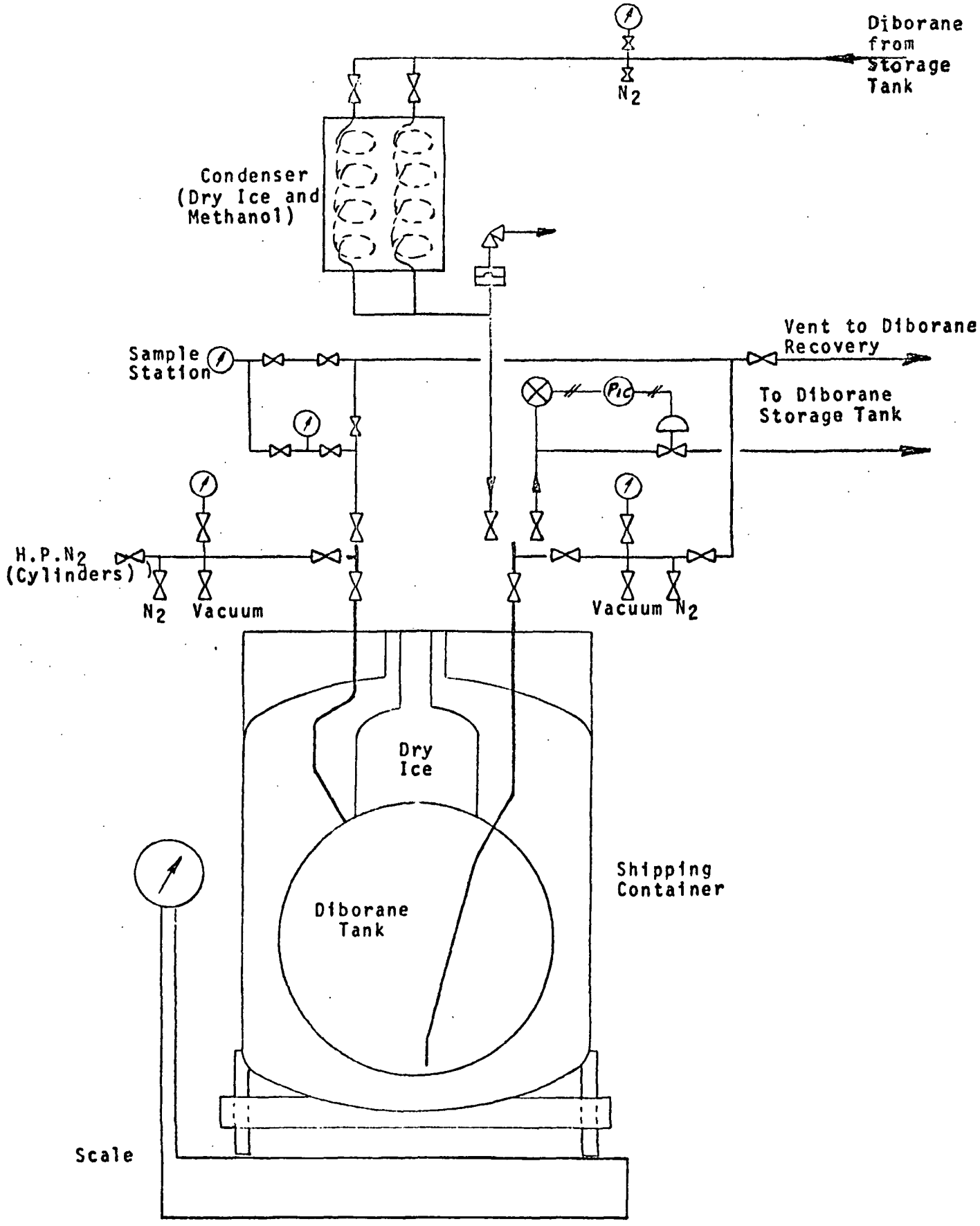


FIGURE 7

LOADING AND UNLOADING FACILITY @ CALLERY
DIBORANE SHIPPING CONTAINER

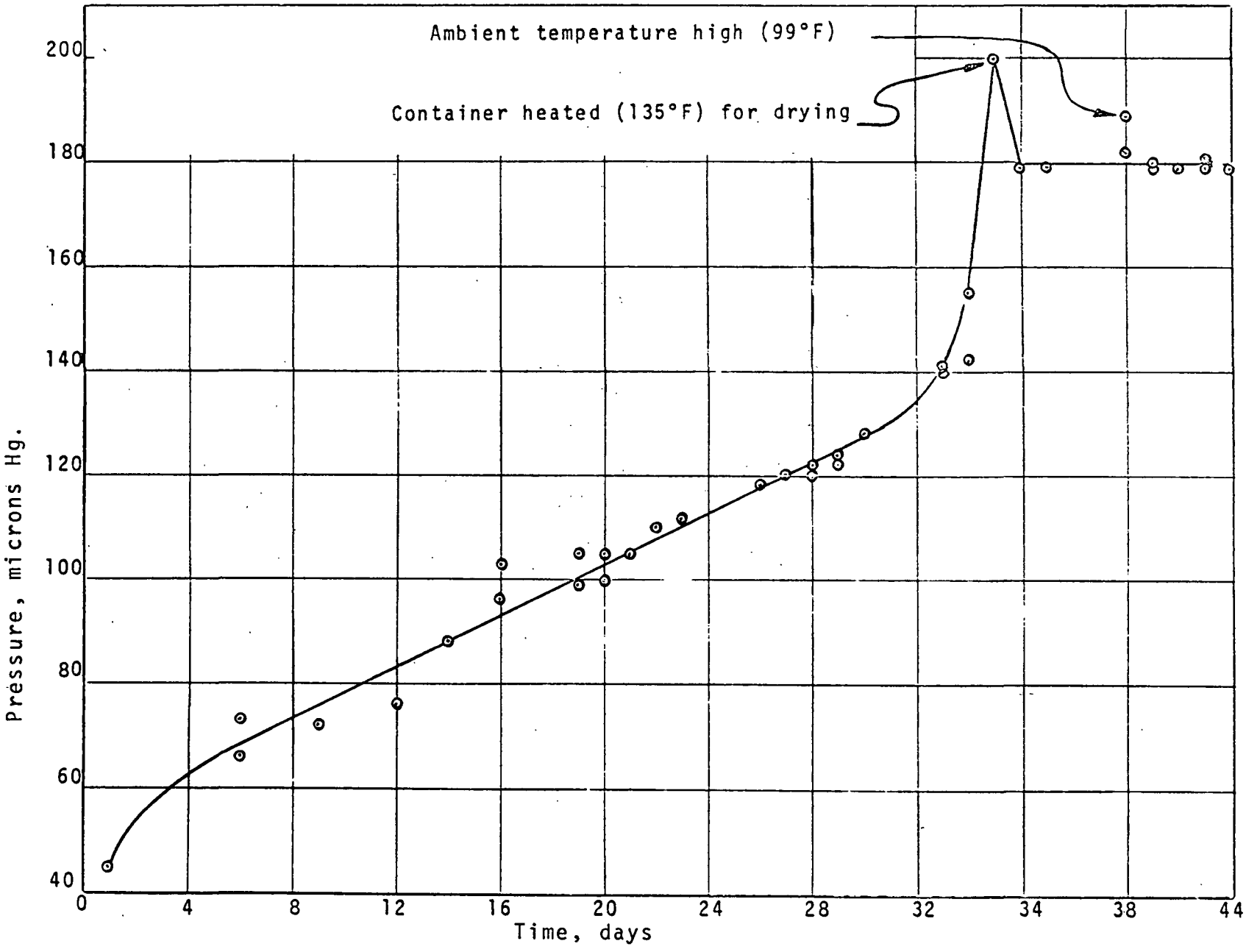


FIGURE 8

VACUUM RETENTION TEST AT CALLERY (DEC. 1969 - JAN. 1970)

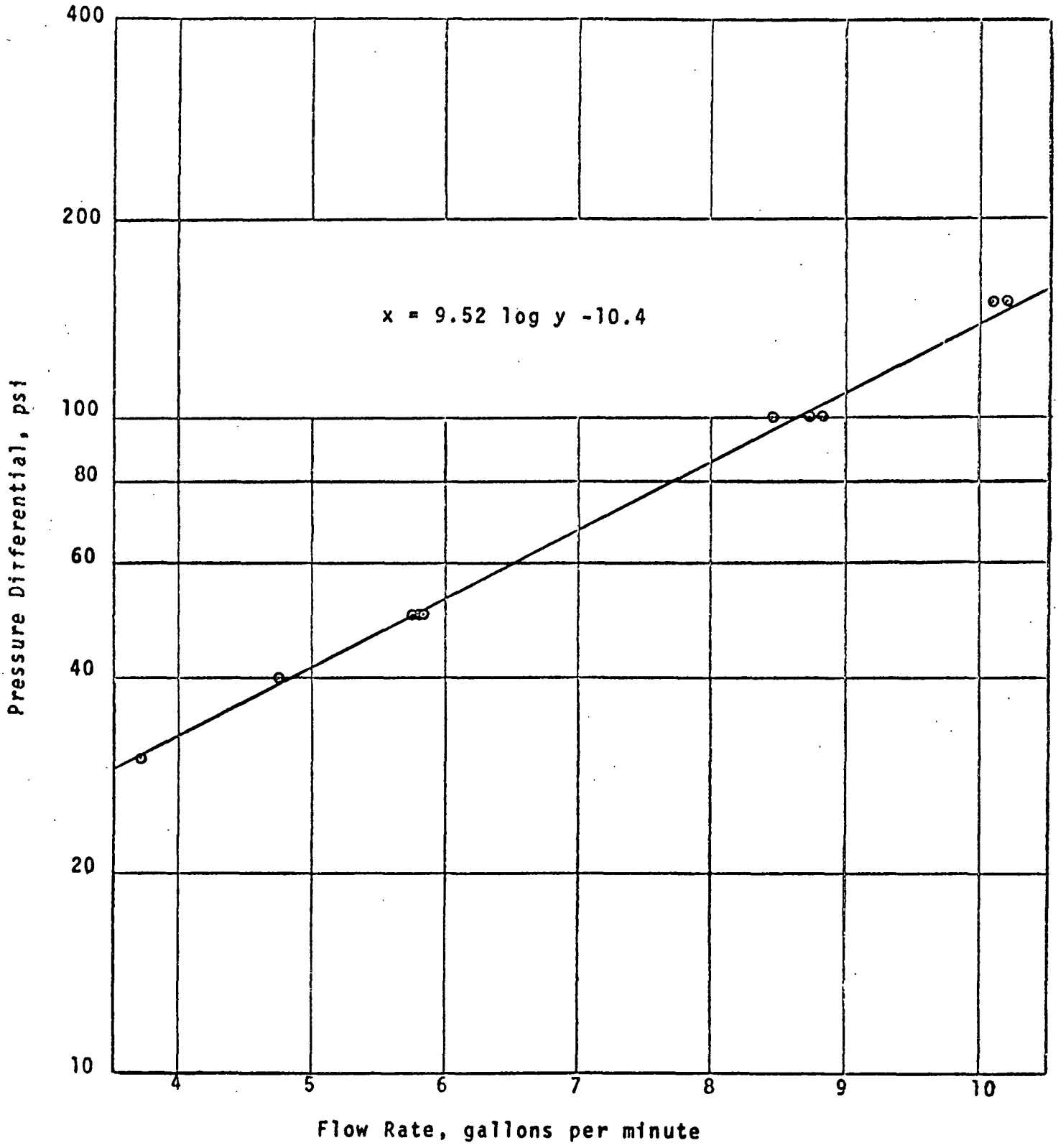


FIGURE 9

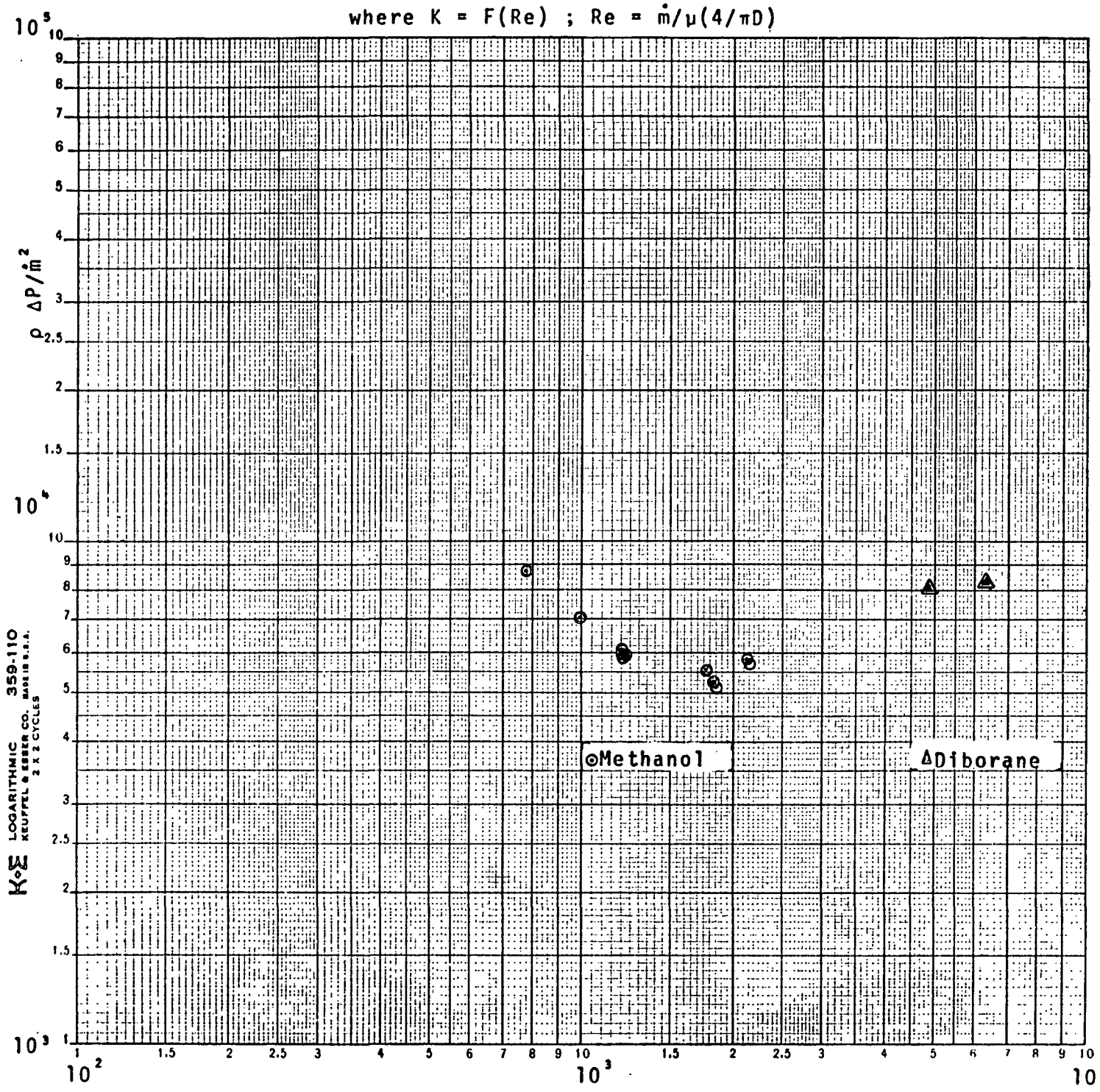
CONTAINER UNLOADING TEST DATA
WITH METHANOL (-1°C.)

FIGURE 9A

CONTAINER UNLOADING DATA

BASED ON $\rho \Delta P/\dot{m}^2 = K/2\, g_c A^2$

where $K = F(Re)$; $Re = \dot{m}/\mu(4/\pi D)$



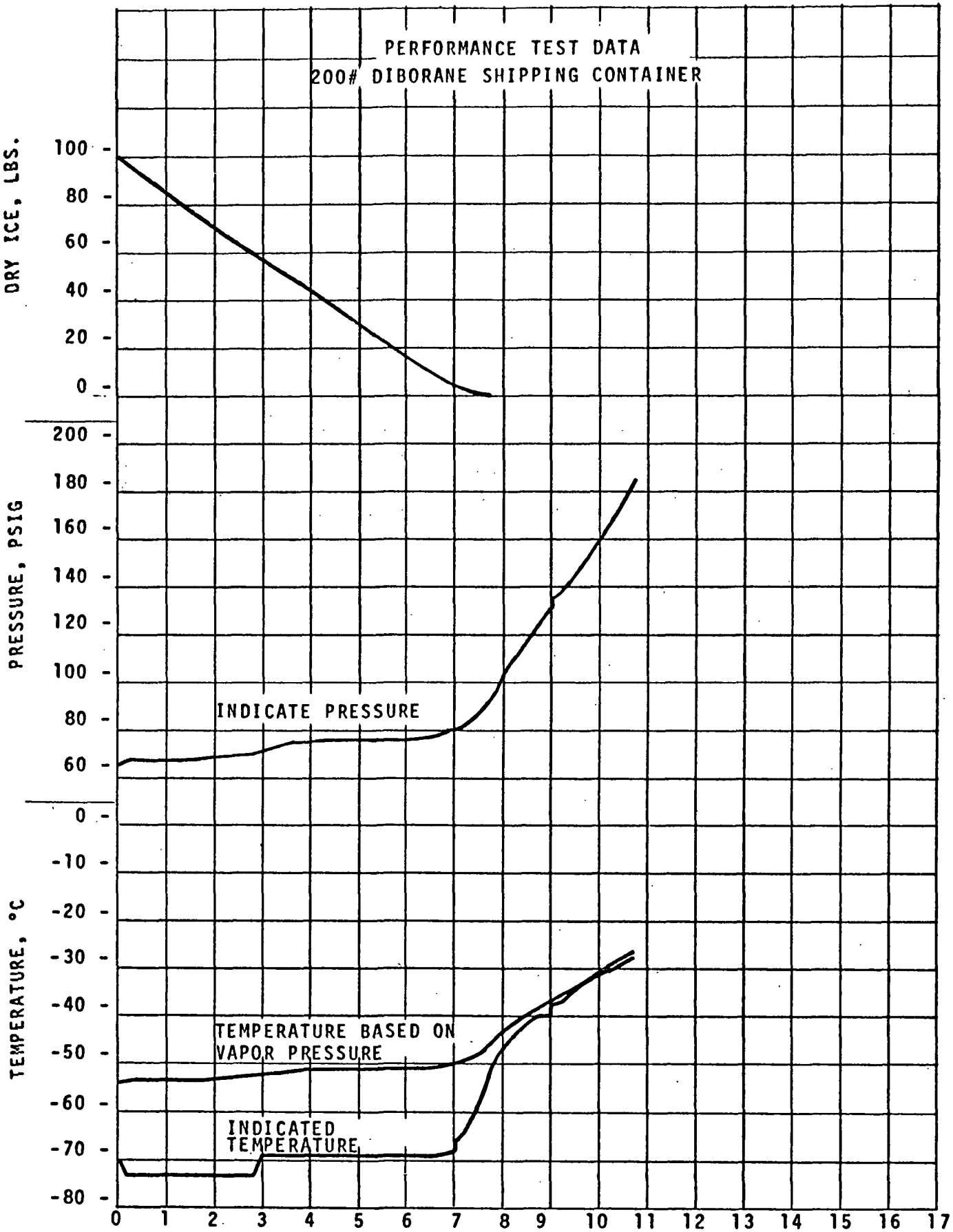


FIGURE 10

PERFORMANCE DATA - TEST # 1

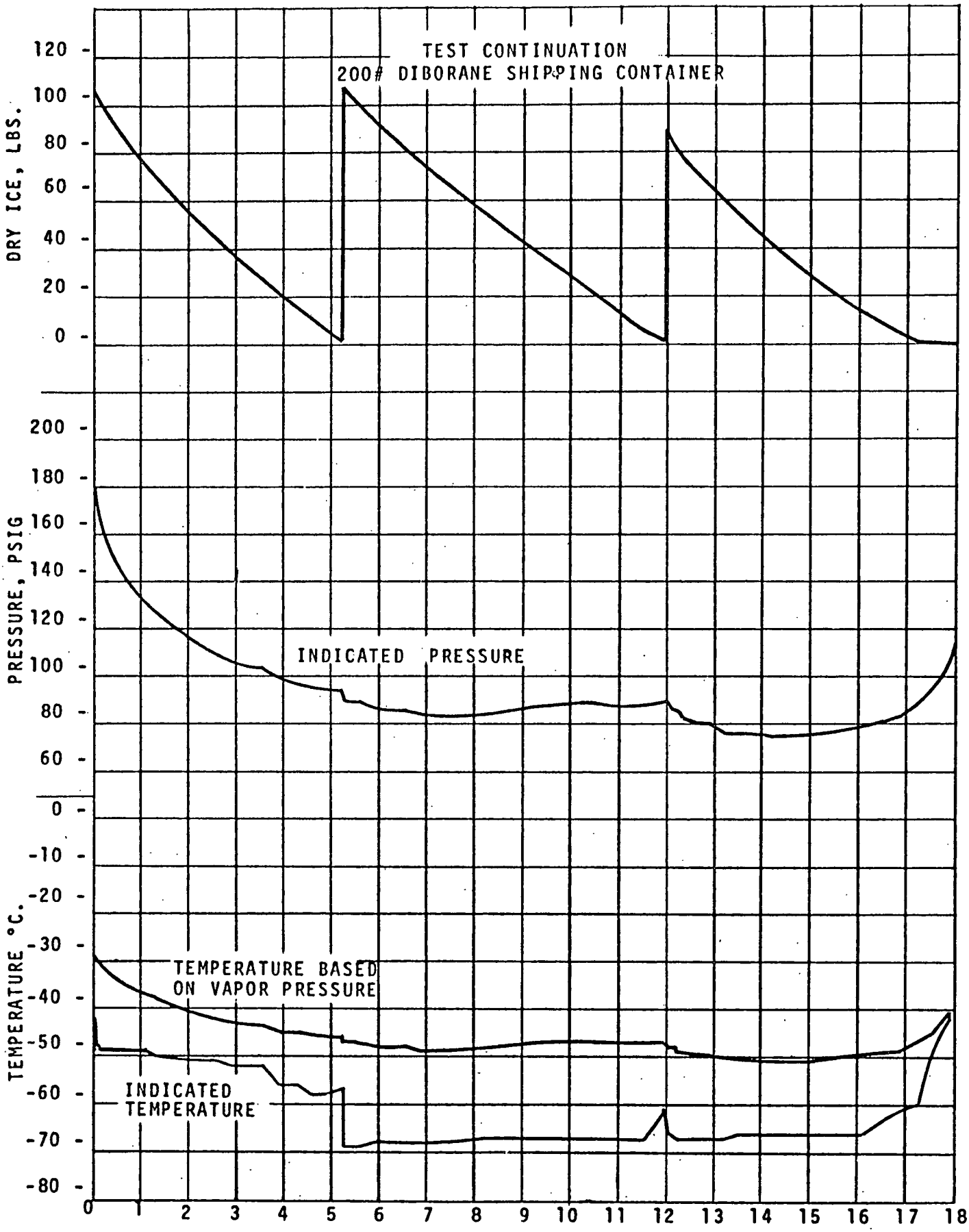


FIGURE 11

CONTINUATION DATA - TEST # 1

PERFORMANCE TEST # 2

CONTAINER MODIFICATIONS

The diborane shipping container, having been thoroughly cleaned, was shipped empty to CVI on 11 August 1970 for modifications. CVI received the container on 25 August 1970, and started evaluation and modifications. The container arrived lying on its side in the truck; however, there was no apparent damage other than a crack in one lucite window.

Upon receipt of the container CVI conducted tests prior to start of modifications. Insulation space vacuum was broken with nitrogen; and a Callery (MSA) borane detector was used to check for diborane in the annular space, with negative results. The vacuum space was evacuated to 15 microns, the inner tank was pressurized with helium to 100 psig, and the entire container was bagged with helium. No leaks were detected by mass spectrometer at maximum sensitivity. This seemed to indicate that there were no leaks; and that vacuum deterioration was due to internal out-gassing from the inner shell, epoxy thread locks, and perlite.

Modifications to the pumpout assembly, bellows protective sleeve, and thermal plug were completed according to details given in the DESIGN AND CONSTRUCTION section.

CONTAINER TESTING AT CVI

The following procedure was used for container testing at CVI:

1. Check perlite fill port; add perlite if necessary.
2. Wrap container with heating tapes and blankets.
3. Pump vacuum while heating for a minimum of 96 hours, or until static vacuum of five microns is achieved. Take periodic vacuum readings three times daily and record.
4. Remove heating tapes, and vacuum retention test for seven days. Record vacuum readings daily.
5. Fill container with liquid nitrogen until liquid level gage indicates half full. Top off daily for three days.
6. Connect gas wet test meter and measure boil-off for eight hours.
7. Drain liquid nitrogen, close vent, and charge dry ice space with 100 pounds of crushed dry ice.

8. Read and record temperature of inner shell daily (same for vacuum) until dry ice is gone.

9. Clean-up and ship.

Vacuum Retention Test

Vacuum retention test data obtained are given in Table 5. Figure 12 shows a plot of data beginning with the minimum pressure achieved (seven microns), and shows a comparison to data obtained on the original pumpdown. This pumpdown was more effective in removing water, believed to be responsible for loss of vacuum in earlier tests.

The vacuum retention test was completed on 10 November 1970. Insulation space pressure rose to nine microns in 20 hours, and remained at nine microns after 75 hours. Later readings of ten microns were obtained, but pressure was down to nine microns when the test was concluded after 310 hours.

Thermal Test with LN_2

Thermal testing was then started by adding liquid nitrogen to the inner tank and dry ice at the top. As expected, the insulation space pressure dropped off scale (less than one micron); but on the morning of the third day pressure was up to ten microns. Helium was charged on top of the LN_2, and a mass spec test was made with negative results. When the LN_2 was gone, the inner tank was pressured to 450 psig with helium; and again a mass spec test was negative. Repeat of the cooling cycle produced the same pressure behavior, sudden rise on the third day. Leakage through the outer shell was then checked by bagging and mass spec tests, and this time the results were positive. The leak was isolated and found to be at the point where the vent tube passes through the outer shell. This joint was rewelded and retested to check that leakage had been stopped.

Following repair of the leak, it was necessary to heatup the inner tank prior to repumping the insulation space. After four days of purging with 250°F. air to heat the inner tank, the exhaust air was still 0°F., insulation space pressure had risen from less than one micron to 6.5 microns. The hot gas purge was continued while pumping the insulation space, which was down to 11 microns on 21 December 1970. Pumpdown was terminated when pressure in the insulation space was down to four microns, with purge gas exit temperature up to 80°F.

Thermal testing was then restarted by filling the inner tank completely full of liquid nitrogen, and packing dry ice in the chamber above the inner tank. Table 6 gives the data obtained, showing an equilibrium heat leak rate less than 85 BTU/hr. Taking into account the lower temperature differential for diborane, calculations indicated a diborane storage capability of 25 to 30 days. On 11 January 1971 CVI was authorized to return the container. Shipment was made on 13 January 1971, and the container arrived at Callery on 21 January 1971.

CHECKOUT AT CALLERY

The container arrived at Callery in good condition. Insulation space pressure remained below five microns for several days, with temperature still below ambient.

Diborane inventory was builtup, and the loading facility was reactived in preparation for charging diborane into the container.

The container was washed with methanol to remove water and/or other reactive materials. Drying was accomplished by pumping vacuum on the inner tank while heating the external section of the inner tank exposed via the dry ice chamber.

Data obtained after receiving the container are given in the beginning of Table 7. Insulation space pressure, which had been less than five microns for about three weeks, rose to 450 microns when the inner tank was heated; but upon subsequent cooling to sub-zero temperatures, the pressure dropped only to about 70 microns. This suggested that heating released hydrogen from the stainless steel and/or released some water vapor from the perlite. Additional cooling was used to distinguish between these effects.

The inner tank was cooled, initially with dry ice and subsequently with liquid nitrogen. Pressure did not drop below 45 microns, which indicated presence of non-condensables, most likely hydrogen released from the stainless steel during heating. It became necessary, therefore, to setup a vacuum system and pump the insulation space to a better vacuum.

REPUMP OF INSULATION SPACE

Data for the repumping are also recorded in Table 7; but are summarized by Figure 13, which shows insulation space pressure and container temperature versus time. The container was reheated as it had been for drying; then the heat was turned off while continuing to pump on the insulation space. The vacuum system initially setup was only capable of achieving 53 microns on the container; so the necessary modifications were made to achieve better vacuum. With the new system, container

pressure was quickly reduced to 15 microns. Pumping was continued for several more days with moderate heating of the container.

PRECOOLING

Diborane production and other preparatory jobs were scheduled with plans to charge diborane into the container the week of 19 April 1971.

Liquid nitrogen was charged to the inner tank on 13 April 1971 to precool the container, allowing one week to reach equilibrium. Procedures were written to prevent any possibility of problems due to the liquid nitrogen precooling. A positive pressure was maintained at all times to prevent condensing water or oxygen in the liquid nitrogen. Additional liquid nitrogen was charged on 16 April 1971; then on 20 April 1971, the day before starting to charge diborane, the excess liquid nitrogen was removed. Thus there was not sufficient liquid nitrogen remaining to cause excessive freezing of the diborane, although the empty inner tank was intentionally cooled to well below the -80°C. at which diborane was to be charged.

Precooling data are given in Table 8. Final temperature after seven days was -100°C.

DIBORANE CHARGE

Diborane was charged into the container on 21 and 22 April 1971. Just prior to charging, the gas inside the inner tank was sampled and found to contain only nitrogen. The charging procedure was designed to achieve an initial liquid diborane temperature approaching -80°C. Data for the diborane charge are given in Table 9.

The diborane was charged in two steps, separated by venting of nitrogen initially present and a small quantity of inerts fed with the diborane. This venting to remove non-condensables is referred to as "topping". Figure 14 shows the main charge, in which 203.5 pounds was added on 21 April 1971. Rate of addition was controlled by a slow drain from the condenser, maintaining sufficient residence time in the condenser for the liquid diborane to approach dry ice temperature. Charging proceeded with no problems; the dip tube plugged momentarily when the liquid reached that height, but the tube freed itself as soon as the feed pressure builtup slightly. This freezing of diborane created no problem, and confirmed that precooling had been effective.

Topping off the non-condensables reduced the contents to 197 pounds, so a small diborane charge was made on 22 April 1971. Afain the container was intentionally overcharged to permit additional topping. Final diborane net weight was 201.5 pounds after the second topping.

THERMAL TEST #2

Immediately after topping to remove most of the non-condensables, dry ice was charged in preparation for the thermal storage test. A total of 108 pounds of dry ice was added, and the container was sealed to begin the test on 1645 on 22 April 1971. The storage test was continued until 21 May 1971. Results were satisfactory in terms of adequate time for shipment.

Complete data for the storage test are given in Table 10. Smoothed data in Table 11 were obtained by plotting dry ice weight, container pressure, and container temperature; and fitting a series of straight lines through each set of data. These points were used to construct the graph of Figure 15. Interpolation and extrapolation of these data produced the even-day results presented in Table 12.

TEST DATA ANALYSIS

To place results in the proper context, it is appropriate to preface the performance data with a consideration of limitations. The limitation on safe storage or shipment time is pressure, as shown by the following table for 200 pounds of diborane.

LIQUID TEMP., °C.	VAPOR PRESSURE, PSIG	LIQUID FILL % of VOL.
0	385	76
9.8	500	88
12.2	550	98
12.5	560	100

Complete liquid fill occurs at 12.5°C.; whereas maximum working pressure of 500 psig is reached near 10°C. For a practical limit, we chose a pressure of 400 psig; which, allowing for small partial pressure of non-condensables, is equivalent to about 0°C. This then becomes the temperature at which the container should be iced to recool; realizing, however, that is not an absolute limit. Secondly, from the product purity standpoint it is desirable that the normal maximum temperature be maintained about -25 to -35°C., below which decomposition is essentially nil. Initially -35°C. had been selected as the normal maximum operating temperature; that is, the temperature reached by a container delivered on schedule. Callery's proposal to NASA stated "The container will be designed and built to maintain liquid diborane.....at temperature between -35°C. and -80°C. for a period of at least 20 days.....". Callery's specification to our sub-contractor, CVI Corporation, had been for 30 days between these temperatures.

Figure 15 and Table 12 show that, although dry ice was spent in fourteen days, it was over twenty days before liquid diborane temperature reached -35°C. When the test was discontinued after 29 days, the pressure was 300 psig and the temperature was -10°C. Extrapolation of temperature rise at the same rate (actually the rate would go down slightly with lower temperature difference) shows that 0°C. would not be reached until over 33 days. Thus Callery's requirement of 20 days to -35°C. was achieved; and this performance was adequate for consideration of a 20-day shipping permit, both in terms of safety factor to locate and reice a lost shipment and in terms of preserving product purity. It should be possible, however, to routinely obtain delivery within fifteen days, particularly in view of the fact that we currently make shipment within ten days. Use of a fifteen day permit may have the effect of expediting delivery more than a twenty day permit. In any case the safe shipping time is well over thirty days.

The insulation space pressure remained low, demonstrating that there was no measureable leak. Absolute pressure was less than one micron at the start of the test, after the liquid nitrogen precooling; and rose to about five microns after fourteen days, when dry ice was gone and diborane temperature neared -70°C. As the temperature rose, insulation space pressure rose to about twenty microns at -10°C.; however, upon recooling to -70°C., pressure returned to about five microns.

Indicated temperature is sensed by a thermocouple on the inside top of the sphere. Proximity of the dry ice influences this temperature reading; therefore, a more accurate temperature for the liquid diborane is obtained from the pressure reading. Using known vapor pressure data, and allowing for partial pressure of the measured non-condensable present, a reliable temperature may be calculated. The temperature reported in Table 12 is the actual temperature, back-calculated from pressure readings. Indicated and actual temperatures began to converge after all of the dry ice was gone.

During the 22nd day the container was lifted and given a turbulent sloshing to check for possible stratification. There was no change at all in the pressure, therefore no change in temperature of the surface layer of liquid with which the vapor is in equilibirium. The indicated temperature did change, supporting the fact that a lag exists because the temperature sensing element is in the vapor space. A longer thermocouple extending to the bottom of the sphere would read liquid diborane temperature and eliminate the lag, therefore would be recommended for future containers.

The alarm activated during the 20th day, when indicated temperature was -32°C. and actual temperature was -40°C.

POST-TEST RECOOL

At the end of the 29th day, dry ice was added. This ended the thermal test to determine safe shipping time; however, data recording was continued to demonstrate the recooling capability of the container.

Data given in Table 13 show that repeated dry ice addition brought the indicated temperature down as low as -77°C. and actual temperature down to -72°C. Diborane could be stored indefinitely under these conditions without any significant decomposition. The temperature indicator required periodic maintenance, having a tendency to stick (for example Table 13, page 74, at 48.97 days); therefore, temperature derived from pressure is referred to as "actual temperature".

On 11 June 1971 the container's center of gravity was experimentally determined by measuring the angle of inclination when the container was suspended from each of the four lifting loops. This information had been requested by DOT, and was included in the Petition to DOT for Special Permit.

On 23 June 1971, after over 61 days from the start of the test, a gas sample was taken to check for possible diborane decomposition. The results showed that diborane decomposition was less than 0.005 percent for a time period of over sixty days.

After taking the above sample, the diborane was removed from the container as needed for various uses.

CONTAINER CLEANOUT

Diborane was removed from the container as demands occurred, and the residual amount was removed from the container back into the regular plant storage tank on 13 August 1971. The container was then given a thorough cleanout. These steps did not present any new operations, having been done after the previous storage test.

After emptying and purging with nitrogen, the container was washed with naphtha, isopropanol, and methanol. No problems were encountered in the cleanout; and no reaction indicative of solids formation was detected, even in the line to the gauge where diborane had been exposed to room temperature for over 100 days.

SHIPPING TEST

The only remaining experimental work required by this program was a shipping test to confirm structural integrity of the container.

Methanol was selected to simulate diborane in the shipping test. The container was to be loaded with 200 pounds of methanol, pressurized with nitrogen, and fully charged with dry ice; and

then shipped to the Callery Chemical Company plant in Lawrence, Kansas. There data were to be recorded, and the container re-shipped back to the Callery, Pennsylvania site.

Methanol wash was removed from the container, and 200 pounds of clean methanol was charged to the container in preparation for the shipping test.

DOT replied to our inquiry about the methanol shipment, and advised that a special permit was required. Permission for test shipment of methanol had to become a part of the diborane special permit. The test shipment was therefore delayed until receipt of the special permit.

To prepare for shipments using the new container, a meeting was held on 25 August 1971 with the terminal manager of the trucking company who handled many of our previous diborane shipments. This meeting, which included our engineering and traffic personnel, was most helpful to inform the carrier of our requirements; and we were pleased by his favorable comments on container design. It appears that the container will not create any special shipping problems.

Upon receipt of the special permit, preparations were made to begin the shipping test. The permit references to Title 49 of the Code of Federal Regulations were obtained and reviewed to assure compliance with the permit. Other shipment preparations were completed, and shipment to Callery's plant in Lawrence, Kansas was made on 12 January 1972 by motor freight.

According to the planned procedure, the container with 200 pounds of methanol was pressurized to 100 psig with nitrogen; and 108 pounds of dry ice was charged into the dry ice chamber just prior to shipment. Insulation space pressure before adding dry ice was 7 microns; however, ambient temperature was low. At 75 to 80°F. temperature, this pressure is about 30 to 40 microns. Gross shipping weight was 2925 pounds.

The container arrived at the Lawrence, Kansas plant on 21 January 1972. There was no apparent damage resulting from the shipment. Thermal data was not part of the shipping test, but the container temperature at arrival was -28°C. Insulation space pressure had dropped to 1.5 microns due to the dry ice added before shipment.

The container was then shipped from the Lawrence, Kansas plant on 28 January 1972. Delivery was made 7 February 1972 to CVI Corporation, where arrangements had been made to correct some inaccuracies in wording on the U-1A Manufacturer's Data Report for Unfired Pressure Vessels. Again there was no apparent

damage in shipment. Container temperature was +1°C., and insulation space pressure was 9 microns.

This successful shipping test completed all experimental work on this program. CVI called the code inspector to correct the U-1A Form, and a Corrected Data Report for Form U-1A Manufacturers' Data Report was issued 21 March 1972. The container was returned to Callery on 18 April 1972, after which it was dried and prepared for service.

TABLE 5

DIBORANE SHIPPING CONTAINER
VACUUM RETENTION TEST @ CVI (NOV. 1970)

DATE	TIME	HOURS	PRESSURE MICRONS Hg	TEMP, °F AMB	TEMP, °F CONTR	REMARKS
10/26/70	1205	0	20	73	-	Stopped pumping
10/26/70	1545	4	23	73	-	
10/27/70	0710	19	20	70	-	
10/27/70	1515	27	19	74	-	
10/28/70	0710	43	18	70	-	
10/28/70	1530	51	14	73	-	
10/29/70	0730	67	14	73	-	
10/29/70	1540	75	10	73	-	
10/30/70	0715	91	10	70	-	
10/30/70	1515	99	9	74	98	
11/2/70	0710	139	9	72	91	
11/2/70	1530	147	7.5	75	89	
11/3/70	0710	163	7.5	72	89	
11/3/70	1100	167	7	74	89	Minimum pressure
11/3/70	1515	171	8	75	88	
11/4/70	0710	187	9	72	88	
11/4/70	1425	194	9	74	88	
11/5/70	0715	211	9	72	88	
11/5/70	1415	218	9	74	88	
11/6/70	0720	235	9	72	86	

TABLE 6

DIBORANE SHIPPING CONTAINER THERMAL TEST WITH LN_2

DATE	TIME	ELAPSED TIME, Hrs	BOILOFF RATE		CUMULATIVE BOILOFF, Ft^3	DEPLETION %	HEAT LEAK BTU/Hr.
			FT^3/Hr.	Lb./Hr.			
12/28/70	1630	0	-	-	-	-	-
	1730	1	77.5	5.45	72.5	0.0	467
	1930	3	42.5	3.10	175	1.8	266
	2230	6	32.5	2.35	280	2.9	202
12/29/70	0130	9	26	1.90	365	3.7	163
	0830	16	20	1.45	-	-	124
	1200	19.5	18.8	1.36	590	6.0	117
	1400	21.5	17.5	1.27	625	6.4	109
	1600	23.5	17.5	1.27	668	6.8	109
	2400	31.5	17.5	1.27	-	-	109
12/30/70	0800	39.5	16.3	1.18	956	9.7	101
	0930	41	15.6	1.13	980	10.0	97
	1330	45	15.2	1.11	1041	10.6	95
1/4/71	0805	159.6	-	-	2571	-	-
	0905	160.6	14.0	1.02	2585	26.3	87
	1010	161.7	13.8	0.99	2600	26.5	84
	1330	165	13.6	0.99	2646	26.9	84
1/5/71	0805	183.6	13.1	0.95	2885	29.4	81
1/6/71	1045	210.2	-	-	-	-	77

TABLE 7

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB TEMP. °F	SCALE READING LBS.	CONTAINER LI %*	CONTAINER VI %*	CONTAINER VACUUM MICRONS	REMARKS
	1/21/71								Container Received
1400	1/22/71	-	-	-	2613	-	-	-	Shipping Weight
1415	1/22/71	-32	22	40	2581	0	76	5.9	Inst. Cover Off
1410	1/25/71	-32	22	32	-	0	60	3.0	
1405	1/28/71	-20	25	10	-	100	45	1.5	
1450	2/1/71	-18	25	2	-	100	45	1.0	
1130	2/3/71	-18	25	17	-	100	42	1.2	
1255	2/5/71	-14	25	50	-	0	62	4.0	Filled W/MeOH
0850	2/8/71	+ 2	0	29	-	100	55	3.5	Drained MeOH
1145	2/8/71	-	-	-	-	-	-	-	Started Vacuum
0820	2/10/71	+ 2	VAC	19	-	0	60	3.0	
0840	2/12/71	+ 2	VAC	40	-	0	64	4.2	
1040	2/15/71	+ 2	VAC	30	-	0	60	3.8	
0950	2/17/71	+45	VAC	60	-	0	68	9.8	Heating to Dry
0815	2/19/71	+82	VAC	70	-	10	70	100	
0815	2/22/71	+99	VAC	79	-	10	70	425	
0910	2/23/71	+99	VAC	64	-	10	70	450	
0845	2/24/71	+60	VAC	75	-	0	70	250	
1500	2/25/71	-22	VAC	72	-	0	70	120	Started Cooling W/Dry Ice
1025	2/26/71	-32	VAC	74	-	0	80	95	
0835	3/1/71	-39	25	72	-	0	80	75	Pressured W/N_2
0825	3/2/71	-50	20	71	-	0	80	70	
0815	3/3/71	-50	20	70	-	0	80	70	
1000	3/4/71	-32	21	65	-	0	75	75	
0830	3/5/71	-48	20	74	-	0	80	71	

*LI = Level Indicator; VI = Voltage Indicator

TABLE 7 (PAGE 2 OF 9)

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F	SCALE READING LBS.	CONTAINER LI %	CONTAINER VI %	CONTAINER VACUUM MICRONS	REMARKS
0825	3/8/71	-61	16	60	-	0	80	66	
0815	3/9/71	-52	20	70	-	0	80	68	
0815	3/10/71	-30	15	60	-	0	80	73	
0830	3/10/71	-	-	-	2578.5	-	-	-	Tare
1331	3/10/71	-15	0	60	2582	*	75	76	Start LN_2 to inner tank
1332	3/10/71	-15	-	-	2582	-	-	68	
1333	3/10/71	-15	-	-	2583	-	-	60	
1336	3/10/71	-15	-	-	2583	-	-	56	
1342	3/10/71	-15	-	-	2583	-	-	54	
1346	3/10/71	-15	-	-	2585	-	-	52	
1349	3/10/71	-15	-	-	2586	-	-	52	
1352	3/10/71	-15	-	-	2586	-	-	51	
1400	3/10/71	-15	-	-	2594	-	-	49	
1405	3/10/71	-15	-	-	2599	-	-	49	
1415	3/10/71	-15	-	-	2605	-	-	49	
1425	3/10/71	-15	-	-	2609	-	-	49	Shut Off LN_2
1455	3/10/71	-15	40	-	2607	-	-	60	
1500	3/10/71	-15	5	-	2603	-	-	60	Start LN_2 to inner tank
1505	3/10/71	-15	-	-	2610	-	-	52	
1515	3/10/71	-15	-	-	2612	-	-	49	LN_2 All In
1652	3/10/71	-20	38	-	2611	-	-	-	Relieving GN2
2400	3/10/71	-39	30	57	2592	-	-	-	Relieving V. Slowly
0810	3/11/71	-51	22	61	2584		80	59	
1300	3/11/71	-52	2	60	2583	-	-	59	Start LN_2 to inner tank
1305	3/11/71	-60	-	-	2585	-	-	47	
1315	3/11/71	-61	-	-	2597	-	-	45	
1330	3/11/71	-61	-	-	2620	-	-	45	
1350	3/11/71	-61	-	-	2638	-	-	45	LN_2 All In
1440	3/11/71	-61	22	58	2636	-	-	46	
1555	3/11/71	-61	35	60	2637	-	-	46	

*Disconnected

TABLE 7 (PAGE 3 OF 9)

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F	SCALE READING LBS.	CONTAINER LI %	CONTAINER VI %	CONTAINER VACUUM MICRONS	VACUUM PUMP, MICRONS	REMARKS
1715	3/11/71	-61	40	61	2635	-	-	46		
2315	3/11/71	-69	40	56	2623	-	-	46		
0810	3/12/71	-79	32	58	2607	-	-	45		
1315	3/12/71	-79	29	62	2600	-	-	45		
1520	3/12/71	-81	28	60	2597	-	-	45		
1530	3/13/71	-81	18	57	2584	-	-	54		
2350	3/14/71	-64	10	59	2583	-	-	61		
0830	3/15/71	-58	9	67	2583	-	-	63		
0815	3/16/71	-42	9	59	2583	-	-	70		
1420	3/16/71	-39	8	57	2582	-	-	72		
1050	3/17/71	-29	5	61	2582	-	-	77		
0810	3/18/71	+39	5	57	-	-	-	130		
0815	3/19/71	+69	8	64	-	-	-	240		
1100	3/19/71	-	-	-	-	-	-	240	40	
1152	3/19/71	-	-	-	-	-	-	240	13	
1250	3/19/71	-	-	-	-	-	-	250	12	
1310	3/19/71	70	9	63	-	-	-	250	11	LN_2 in trap
1314	3/19/71	-	-	-	-	-	-	250	11	Start open to Container
1315	3/19/71	-	-	-	-	-	-	-	12.5	
1316	3/19/71	-	-	-	-	-	-	245	13	Wide open to Container
1318	3/19/71	-	-	-	-	-	-	-	14	
1320	3/19/71	70	-	-	-	-	-	240	15	
1355	3/19/71	70	-	-	-	-	-	195	15	
1450	3/19/71	70	9	68	-	-	-	169	13	
1520	3/19/71	71	-	-	-	-	-	157	13	
1620	3/19/71	72	-	-	-	-	-	143	11.5	
1715	3/19/71	-	-	-	-	-	-	135	11	
1915	3/19/71	76	-	-	-	-	-	127	10	
2145	3/19/71	78	-	-	-	-	-	123	10	
2400	3/19/71	80	-	-	-	-	-	123	10	Pump off
1700	3/20/71	90	7	67	-	-	-	210	Atm.	

TABLE 7 (PAGE 4 OF 9)

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F	SCALE READING LBS.	CONTAINER LI %	CONTAINER VI %	CONTAINER VACUUM MICRONS	VACUUM PUMP, MICRONS	REMARKS
1545	3/21/71	97	6	65	-	-	-	330	Atm.	
0830	3/22/71	99	6	63	-	-	-	430	Atm.	
0907	3/22/71	-	-	-	-	-	-	380	38	Cont. Valve Closed
0920	3/22/71	99	-	-	-	-	-	390	17	
0933	3/22/71	-	-	-	-	-	-	390	10.5	Start LN_2 to Trap
0935	3/22/71	-	-	-	-	-	-	380	11	Cont. Valve Open
0939	3/22/71	-	-	-	-	-	-	370	12.5	
1024	3/22/71	99	6	71	-	-	-	330	12.5	
1118	3/22/71	99	-	-	-	-	-	300	11	Add more LN_2
1124	3/22/71	99	-	-	-	-	-	300	10.5	LN_2 Trap Full
1224	3/22/71	99	-	-	-	-	-	285	10	
1324	3/22/71	99	-	-	-	-	-	275	10	
1330	3/22/71	99	-	72	-	-	-	285	10.5	
1455	3/22/71	99	-	72	-	-	-	285	10.0	Cont. Valve Closed
1530	3/22/71	99	-	70	-	-	-	285	10.0	
1555	3/22/71	99	-	72	-	-	-	300	10.0	
1624	3/22/71	99	-	63	-	-	-	295	10.0	
1638	3/22/71	99	-	65	-	-	-	295	10.0	Opened Contr Shutoff Heat
1648	3/22/71	99	-	67	-	-	-	290	10.0	
1728	3/22/71	99	-	63	-	-	-	280	9.8	
1915	3/22/71	92	-	67	-	-	-	260	9.8	
2100	3/22/71	88	-	69	-	-	-	240	9.8	
2400	3/22/71	83	-	69	-	-	-	220	9.8	Closed Contr; Shut-off Vac.
0835	3/23/71	73	-	69	-	-	-	195	Atm.	
0910	3/23/71	72	-	72	-	-	-	190	Atm.	
0950	3/23/71	72	-	61	-	-	-	192	Atm.	
1010	3/23/71	71	-	69	-	-	-	183	11	Vac on; Contr Open
1130	3/23/71	70	-	69	-	-	-	168	11	
1300	3/23/71	69	-	71	-	-	-	158	10.5	
1345	3/23/71	69	-	72	-	-	-	155	10.0	
1430	3/23/71	68	-	73	-	-	-	149	10.0	

TABLE 7 (PAGE 5 OF 9)

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F	SCALE READING LBS.	CONTAINER LI %	CONTAINER VI %	CONTAINER VACUUM MICRONS	VACUUM PUMP, MICRONS	REMARKS
1600	3/23/71	67	-	63	-	-	-	143	10.0	
1715	3/23/71	66	-	67	-	-	-	138	9.8	
1915	3/23/71	61	-	66	-	-	-	131	9.8	
2145	3/23/71	60	-	69	-	-	-	122	9.8	
2400	3/23/71	59	-	63	-	-	-	116	9.8	
0835	3/24/71	53	-	65	-	-	-	98	11	(10 After LN_2 Added)
1000	3/24/71	52	-	74	-	-	-	95	10	
1200	3/24/71	52	-	65	-	-	-	92	9.8	
1300	3/24/71	52	-	63	-	-	-	90	9.8	
1500	3/24/71	51	-	59	-	-	-	87.5	9.8	
1630	3/24/71	50	-	67	-	-	-	86	9.8	Added LN2 @ 1730
1915	3/24/71	50	-	65	-	-	-	99	12	
2030	3/24/71	50	-	65	-	-	-	98	11	
2200	3/24/71	49	-	62	-	-	-	94	10.2	
2400	3/24/71	48	-	61	-	-	-	89	9.8	
0755	3/25/71	-	-	-	-	-	-	78	11	
0815	3/25/71	-	-	-	-	-	-	76	10	LN_2 Added
0915	3/25/71	45	-	61	-	-	-	Inst. Stuck		
1320	3/25/71	44	-	62	-	-	-	73	9.7	
1445	3/25/71	43	-	-	-	-	-	77	11	
1600	3/25/71	42.5	-	61	-	-	-	77	10.3	
1915	3/25/71	41.5	-	67	-	-	-	75	-	
2100	3/25/71	41	-	69	-	-	-	74	-	
0015	3/26/71	40.5	-	69	-	-	-	72	-	
0800	3/26/71	39	-	-	-	-	-	68	-	
0845	3/26/71	39	-	62	-	-	-	67.5	9.8	
1115	3/26/71	-	-	-	-	-	-	66	10.0	Added LN_2 Added
1145	3/26/71	39	-	63	-	-	-	66	10.0	
1445	3/25/71	38	-	63	-	-	-	65.5	10.0	
1515	3/26/71	-	-	-	-	-	-	65.5	9.75	
1915	3/26/71	37	-	62	-	-	-	65.5	10→9.7	LN_2 Added
2400	3/26/71	37	-	62	-	-	-	65	9.8	

TABLE 7 (PAGE 6 OF 9)

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F	SCALE READING LBS.	CONTAINER LI %	CONTAINER VI %	CONTAINER VACUUM MICRONS	VACUUM PUMP, MICRONS	REMARKS
0830	3/27/71	36	-	65	-	-	-	62.5	9.7	
1300	3/27/71	35	-	67	-	-	-	61	10.0	
1440	3/27/71	35	-	67	-	-	-	60.5	9.9	
1530	3/27/71	35	-	66	-	-	-	65	11.5	
1540	3/27/71	-	-	-	-	-	-	64.5	9.7	
1800	3/27/71	35	-	63	-	-	-	63	9.9	
0015	3/28/71	-	-	-	-	-	-	61	9.75	
0815	3/28/71	33	-	71	-	-	-	59	9.8 → 9.5	
1330	3/28/71	32.5	-	69	-	-	-	60.5	10.3	
1600	3/28/71	32.5	-	71	-	-	-	61	11	
1800	3/28/71	32	-	71	-	-	-	61.5	10.5	
1915	3/28/71	32	-	69	-	-	-	61	10.5	
2015	3/28/71	32	-	-	-	-	-	61	10.2	
2045	3/28/71	-	-	-	-	-	-	60	10.0	
2050	3/28/71	-	-	-	-	-	-	59.8	10.0	
0015	3/29/71	32	-	63	-	-	-	59.5	10.0	
0830	3/29/71	31.5	-	59	-	-	-	57	9.8	
1115	3/29/71	30	-	-	-	-	-	59*	10	
1130	3/29/71	-	-	-	-	-	-	57	-	Vacuum pump off
1315	3/29/71	-	-	-	-	-	-	57	-	Pump oil changed
1330	3/29/71	-	-	-	-	-	-	-	3.7	
1430	3/29/71	30	-	62	-	-	-	59*	15	Open to cont'r.
1645	3/29/71	30	-	62	-	-	-	58	9.6	
1915	3/29/71	29.5	-	63	-	-	-	55.5	9.4	
0015	3/30/71	29.5	-	57	-	-	-	54.5	9.4	
0845	3/30/71	29	-	65	-	-	-	53	9.5 → 9.1	
1015	3/30/71	30	-	60	-	-	-	54	9.5	
1330	3/30/71	29	-	64	-	-	-	54	9.7 → 9.5	
1645	3/30/71	29	-	56	-	-	-	53	9.2	
1900	3/30/71	28.5	-	61	-	-	-	53	9.4	
2400	3/30/71	28.5	-	62	-	-	-	53	9.1	

TABLE 7 (PAGE 7 OF 9)

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F	SCALE READING LBS.	CONTAINER LI %	CONTAINER VI %	CONTAINER VACUUM MICRONS	VACUUM PUMP, MICRONS	REMARKS
0755	3/31/71	27	-	-	-	-	-	52	9.0	
0830	3/31/71	-	-	59	-	-	-	52.5	9.5	
0915	3/31/71	27.5	-	57	-	-	-	52.5	9.6	
1315	3/31/71	27.5	-	64	-	-	-	53	9.7	
1420	3/31/71	29	-	65	-	-	-	54	9.5	
1600	3/31/71	27.5	-	67	-	-	-	54	9.5	
1700	3/31/71	27.5	-	67	-	-	-	55	9.8	
2130	3/31/71	27	-	63	-	-	-	54	9.3	
2400	3/31/71	27	-	62	-	-	-	53	9.5 → 9.2	
0815	4/1/71	27	-	69	-	-	-	52.5	9.5	
1345	4/1/71	27	-	73	-	-	-	56	10.5	Closed to cont'r.
1515	4/1/71	-	-	-	-	-	-	56	-	
1555	4/1/71	27	-	64	-	-	-	55	15	
1600	4/1/71	27	-	-	-	-	-	55	16	Open to cont'r.
1645	4/1/71	27	-	69	-	-	-	65.5	16.5	
1715	4/1/71	-	-	-	-	-	-	68	16	
1730	4/1/71	-	-	-	-	-	-	69	15.5	
1915	4/1/71	26.5	-	69	-	-	-	69	15	
2400	4/1/71	26	-	69	-	-	-	66	14.0	
0755	4/2/71	26	-	-	-	-	-	61	14	
0815	4/2/71	27	-	62	-	-	-	61	14	
1145	4/2/71	-	-	-	-	-	-	61	13.5	
1400	4/2/71	26	-	67	-	-	-	61.5	13	
1715	4/2/71	26	-	67	-	-	-	60.5	13	
2100	4/2/71	26	-	63	-	-	-	60	13	
2400	4/2/71	26	-	61	-	-	-	59.5	13	
0830	4/3/71	26	-	61	-	-	-	58	12.5	
1300	4/3/71	26	-	67	-	-	-	59	13	
1800	4/3/71	26	-	67	-	-	-	58	12.5	
0815	4/4/71	26	-	57	-	-	-	57.5	13	
1330	4/4/71	26	-	61	-	-	-	57.5	14.5	
1915	4/4/71	26	-	65	-	-	-	57	14.5	

TABLE 7 (PAGE 8 OF 9)

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F.	SCALE READING LBS.	CONTAINER LI %	CONTAINER VI %	CONTAINER VACUUM MICRONS	VACUUM PUMP, MICRONS	REMARKS
0100	4/5/71	26	-	59	-	-	-	56.5	14.5	
0810	4/5/71	25	-	58	-	-	-	56	14	
0840	4/5/71	-	-	-	-	-	-	55.5	14.5	Close to cont'r.
1410	4/7/71	25	-	62	-	-	-	54.5	5	Cont'r. closed
1415	4/7/71	-	-	-	-	-	-	52.5	7	Cont'r. open
1515	4/7/71	23	-	63	-	-	-	36	5.3	
1630	4/7/71	23	-	63	-	-	-	24	4.3	
1700	4/7/71	-	-	-	-	-	-	22	-	
1730	4/7/71	-	-	-	-	-	-	20	4.0	
1915	4/7/71	-	-	-	-	-	-	16.5	3.9	
2000	4/7/71	-	-	-	-	-	-	15.5	3.8	
2015	4/7/71	-	-	-	-	-	-	15.0	-	
2115	4/7/71	-	-	-	-	-	-	14.5	3.7	
2215	4/7/71	-	-	-	-	-	-	14.5	3.8	
2400	4/7/71	-	-	-	-	-	-	14.0	4.0	
0810	4/8/71	23	-	58	-	-	-	14.5	5.0	
0815	4/8/71	-	-	-	-	-	-	14.5	5.1	
0900	4/8/71	23	-	57	-	-	-	14.0	4.2	
0945	4/8/71	-	-	-	-	-	-	-	-	
1115	4/8/71	30	-	-	-	-	-	15.0	-	
1400	4/8/71	47.5	-	-	-	-	-	18.0	4	
1545	4/8/71	51	-	69	-	-	-	21.0	4.1	
1600	4/8/71	52	-	-	-	-	-	22	4	
1720	4/8/71	51	-	-	-	-	-	20	4.0	
1723	4/8/71	-	-	-	-	-	-	20	-	
1915	4/8/71	48	-	-	-	-	-	21	-	
0830	4/9/71	33	-	61	-	-	-	19	4	
0833	4/9/71	-	-	-	-	-	-	19	4.5	
1130	4/9/71	47	-	75	-	-	-	23	4.5	
1230	4/9/71	47	-	77	-	-	-	23	4.2	
1400	4/9/71	47	-	79	-	-	-	23	4.1	
1630	4/9/71	43	-	80	-	-	-	25.5	3.9	

TABLE 7 (PAGE 9 OF 9)

DIBORANE SHIPPING CONTAINER - PRETEST DATA

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	AMB. TEMP. °F	SCALE READING LBS.	CONTAINER LI %	CONTAINER VI %	CONTAINER VACUUM MICRONS	VACUUM PUMP, MICRONS	REMARKS
0820	4/12/71	30	-	58	-	-	-	19	3.5	
0823	4/12/71	-	-	-	-	-	-	18.5	3	
1400	4/12/71	30	-	83	-	-	-	23	4.0	
1700	4/12/71	30	-	85	-	-	-	25	4.3	
1900	4/12/71	30	-	80	-	-	-	25	3.8	
2100	4/12/71	30	-	74	-	-	-	24	3.0	
2230	4/12/71	30	-	69	-	-	-	22.5	2.4	
2400	4/12/71	30	-	67	-	-	-	21.5	2.0	
0820	4/13/71	30	-	71	-	-	-	21	-	
1020	4/13/71	31	200	78	-	-	-	22	3.5	
1305	4/13/71	31	0	82	-	-	-	24	3.5	

TABLE 8 (PAGE 1 OF 2)

DIBORANE SHIPPING CONTAINER - PRECOOLING

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	CONTAINER VACUUM MICRONS	AMBIENT TEMP. °F.	SCALE READING LBS.	LN_2 WEIGHT LBS.	REMARKS
1320	4/13/71	29	-	24.5	-	2582.5	0	Started adding LN_2
1326	4/13/71	29	-	7.0	-	2583.5	1	
1328	4/13/71	29	-	5.5	-	2584	1.5	
1335	4/13/71	29	-	4.0	-	2585	2.5	
1350	4/13/71	29	-	3.5	-	2589	6.5	
1410	4/13/71	26	-	3.0	-	2603	20.5	
1425	4/13/71	25	-	2.9	-	2608	25.5	LN_2 off
1450	4/13/71	21	-	6.0	-	2602	19.5	LN_2 on
1503	4/13/71	20	-	3.0	-	2611	28.5	Compl 1st 50 l.
1505	4/13/71	-	-	-	-	-	-	Start 2nd 50 l.
1540	4/13/71	19	-	2.5	-	2678	95.5	Compl 2nd 50 l.
1542	4/13/71	-	-	-	-	-	-	Start 3rd 50 l. (half full)
1550	4/13/71	19	-	2.3	-	2695	112.5	Compl 3rd 50 l.
1605	4/13/71	14	19	16	-	2691	108.5	
1625	4/13/71	-	-	23	-	-	-	
0810	4/14/71	-60	30	0.5	50	2648	65.5	
0915	4/14/71	-62	30	0.8	53	2647	64.5	
1300	4/14/71	-70	30	0.9	58	2640	57.5	
1650	4/14/71	-74	29	0.6	60	2634	51.5	
2200	4/14/71	-80	29	0.1	56	2627	44.5	
0815	4/15/71	-89	30	0	50	2614	31.5	
0920	4/15/71	-88	29	0.3	55	2613	30.5	
1340	4/15/71	-88	29	0.9	65	2608	25.5	
1650	4/15/71	-88	29	0.8	63	2604	21.5	
2330	4/15/71	-88	28	0.2	54	2598	15.5	
0810	4/16/71	-90	30	0	54	2591	8.5	
0915	4/16/71	-90	8	0	56	2795	212.5	LN_2
1200	4/16/71	-92	18	1.0	67	2793	210.5	
1625	4/16/71	-93	29	1.4	69	2793	210.5	
1200	4/17/71	-100	39	0.3	60	2781	198.5	
2245	4/17/71	-101	39	0.2	59	2771	188.5	

TABLE 8 (PAGE 2 OF 2)

DIBORANE SHIPPING CONTAINER - PRECOOLING

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	CONTAINER VACUUM MICRONS	AMBIENT TEMP. °F.	SCALE READING LBS.	LN2 WEIGHT LBS.	REMARKS
1320	4/18/71	-103	39	1.1	70	2754	171.5	
0830	4/19/71	-103	38	0.4	61	2732	149.5	
0905	4/19/71	-102	40	0.5	63	2731	148.5	
1545	4/19/71	-102	38	2.1	79	2723	140.5	
2200	4/19/71	-103	38	1.0	65	2716	133.5	
0810	4/20/71	-102	40	0.2	57	2705	122.5	
1115	4/20/71	-103	75	2.8	70	2704	121.5	Leak Testing
1345	4/20/71	-102	205	1.7	73	-	-	Removed LN2
1620	4/20/71	-102	5	2.0	78	2588.5	-	
2000	4/20/71	-102	5	1.7	71	2588	-	
0805	4/21/71	-100	5	0.5	60	2588	-	

TABLE 9 (PAGE 1 OF 5)

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	CONTAINER VI* %	CONTAINER LI* %	CONTAINER VAC	COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B_2H_6 WEIGHT LBS	CHARGE TIME MIN.	REMARKS
0805	4/21/71	-100	5	-	0	0.5	-	60	-	-	-	Gas sample 1
0905	4/21/71	-	-	-	-	-	-	-	2580	-	-	Tare disconnected
1000	4/21/71	-	-	-	-	-	-	-	2600	-	-	Tare connected
1105	4/21/71	-98	0	-	-	0.6	-	61	2617	-	-	Dry ice added
1129	4/21/71	-	0	-	-	-	110	-	-	-	-	Open to cond.
1132	4/21/71	-98	0	80	0	0.4	-	59	2617	-	-	
1144	4/21/71	-	0	-	-	-	110	-	2617	0	0	Started charge
1146	4/21/71	-	10	-	-	-	-	-	2618	1	2	
1147	4/21/71	-98	10.5	-	-	-	-	-	2620	3	3	
1148	4/21/71	-	10.5	-	-	-	-	-	2622	5	4	
1150	4/21/71	-	10.5	-	-	-	101	-	2624	7	6	
1151	4/21/71	-	11	-	-	-	-	-	2625	8	7	
1153	4/21/71	-	11	-	-	-	96	-	2625	8	9	Dip tube plug
1156	4/21/71	-	11	-	-	-	82	-	2627	10	12	Tube opened
1157	4/21/71	-	11	-	-	-	84	-	2628	11	13	
1157.5	4/21/71	-98	12	-	-	-	84	-	2630	13	13.5	
1159	4/21/71	-98	13	80	7	-	90	-	2633	16	15	
1200	4/21/71	-98	-	-	-	-	-	-	2634	17	16	Closed B_2 feed
1233	4/21/71	-97	-	-	-	-	126	57	2635	18	-	
1241	4/21/71	-	14	-	-	-	122	-	2635	18	16	Opened B_2 feed
1242	4/21/71	-	-	-	-	-	-	-	2636	19	17	
1243	4/21/71	-	14	-	-	0.4	-	-	2638	21	18	
1243.5	4/21/71	-	-	-	-	-	-	-	2639	22	18.5	Reduced feed rate
1244	4/21/71	-	14	-	-	-	-	-	2641	24	19	
1245	4/21/71	-	16	-	-	-	-	-	2642	25	20	
1246	4/21/71	-	-	-	-	-	-	-	2644	27	21	
1248	4/21/71	-97	19	-	15	-	-	-	2646	29	23	
1249	4/21/71	-	-	-	-	-	-	-	2648	31	24	Reduced feed rate
1250	4/21/71	-	-	-	-	-	104	-	2649	32	25	
1251	4/21/71	-	20	-	-	-	-	-	2650.5	33.5	26	
1253	4/21/71	-	20	-	-	-	-	-	2652.5	35	28	
1255	4/21/71	-	-	-	-	-	-	-	2653	36	30	
1257.5	4/21/71	-	-	-	-	-	-	-	2655	38	32.5	

*LI = Level Indicator; VI = Voltage Indicator

TABLE 9 (PAGE 2 OF 5)

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	CONTAINER VI %	CONTAINER LI %	CONTAINER VAC	COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B_2H_6 WEIGHT LBS	CHARGE TIME MIN.	REMARKS
1259	4/21/71	-	20	-	-	-	-	-	2657	40	34	
1301	4/21/71	-	-	-	-	-	98	-	2659	42	36	
1304	4/21/71	-96	20	-	21	-	-	-	2661	44	39	
1306	4/21/71	-	20	-	-	-	-	-	2663	46	41	
1308	4/21/71	-	21	-	-	-	-	-	2665	48	43	
1311	4/21/71	-	21	-	-	-	94	-	2667	50	46	
1313	4/21/71	-96	21	-	-	-	94	-	2669	52	48	
1316	4/21/71	-	22	-	-	-	-	-	2671	54	51	
1318	4/21/71	-	24	-	-	-	-	-	2673	56	53	
1321	4/21/71	-95	24	-	-	-	90	-	2675	58	56	
1323	4/21/71	-	-	-	-	-	-	-	2677	60	58	
1325	4/21/71	-	25	-	-	-	87	-	2679	62	60	
1328	4/21/71	-	27	-	-	-	-	-	2681	64	63	
1331	4/21/71	-	27	-	-	-	86	-	2683	66	66	
1333	4/21/71	-	-	-	-	-	-	-	2685	68	68	
1336	4/21/71	-	30	-	30	-	-	-	2687	70	71	
1338	4/21/71	-95	30	-	-	-	84	-	2689	72	73	
1340.5	4/21/71	-	30	-	-	-	-	-	2691	74	75.5	
1345	4/21/71	-	30	-	-	-	-	-	2695	78	80	
1348	4/21/71	-	30	-	-	-	-	-	2697	80	83	
1350.5	4/21/71	-	-	-	-	-	87	-	2699	82	85.5	
1353	4/21/71	-	-	-	-	-	-	-	2701	84	88	
1355	4/21/71	-	-	-	-	-	-	-	2703	86	90	
1358	4/21/71	-95	31	-	35	-	92	-	2705	88	93	
1359.5	4/21/71	-	31	-	-	-	-	-	2707	90	94.5	
1401	4/21/71	-	31	-	-	-	-	-	2709	92	96	
1403.5	4/21/71	-	-	-	-	-	93	-	2711	94	98.5	
1406	4/21/71	-	32	-	-	-	-	-	2713	96	101	
1408.5	4/21/71	-	-	-	-	-	-	-	2715	98	103.5	
1411	4/21/71	-	35	-	-	-	93	-	2717	100	106	
1413	4/21/71	-95	35	-	38	-	-	-	2719	102	108	
1415	4/21/71	-95	35	-	-	-	93	-	2721	104	110	

TABLE 9 (PAGE 3 OF 5)

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	CONTAINER TI °C	CONTAINER PI PSIG	CONTAINER VI %	CONTAINER LI %	CONTAINER VAC μ	COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B_2H_6 WEIGHT LBS	CHARGE TIME MIN.	REMARKS
1417	4/21/71	-	37	-	-	-	-	-	2723	106	112	
1419	4/21/71	-	37	-	-	-	95	-	2725	108	114	
1421	4/21/71	-	38	-	-	0.2	-	55	2727	110	116	
1423	4/21/71	-	39	-	-	-	-	-	2729	112	118	
1426	4/21/71	-	39	-	-	-	98	-	2731	114	121	
1428	4/21/71	-	40	-	43	-	-	-	2733	116	123	
1430	4/21/71	-	40	-	-	-	-	-	2735	118	125	
1431.5	4/21/71	-	40	-	-	-	100	-	2737	120	126.5	
1433.5	4/21/71	-	40	-	-	-	-	-	2739	122	128.5	
1436	4/21/71	-	40	-	-	-	98	-	2741	124	131	
1438	4/21/71	-	40	-	-	-	-	-	2743	126	133	
1440	4/21/71	-	40	-	-	-	-	-	2745	128	135	
1443	4/21/71	-	40	-	-	-	-	-	2747	130	138	
1445	4/21/71	-	40	-	-	-	-	-	2749	132	140	
1506	4/21/71	-91	43	-	51	0.2	-	55	2767.5	150.5	161	
1509	4/21/71	-	43	-	-	-	-	-	2770	153	164	
1515	4/21/71	-91	45	-	-	-	82	-	2775	158	170	
1522	4/21/71	-91	46	-	-	-	81	-	2780	163	176	
1528	4/21/71	-91	47	-	-	-	78	-	2785	168	182	
1535	4/21/71	-91	48	-	-	-	76	-	2790	173	189	
1540	4/21/71	-91	49	-	60	-	75	-	2793	176	194	
1544	4/21/71	-91	49	-	-	-	72	-	2796	179	198	
1550	4/21/71	-	50	-	-	-	71	-	2798.5	181.5	204	
1552	4/21/71	-	50	-	-	-	-	-	2800	183	206	
1554	4/21/71	-91	50	-	-	0.2	69	56	2801	184	208	
1558	4/21/71	-91	50	-	-	-	68	-	2803	186	212	
1601	4/21/71	-91	50	-	-	-	65	-	2805	188	215	
1603	4/21/71	-	50	-	-	-	-	-	2806	189	217	
1606	4/21/71	-90.5	50	-	-	-	64	-	2807	190	220	
1609	4/21/71	-90.5	50	-	-	-	-	-	2809	192	223	
1612	4/21/71	-90	50	-	-	-	64	-	2810	193	226	
1618	4/21/71	-90.5	50	-	-	-	62	-	2812	195	232	

TABLE 9 (PAGE 4 of 5)

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	TI °C	PI PSIG	VI %	LI %	VAC	COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B_2H_6 WEIGHT LBS	CHARGE TIME MIN.	REMARKS
1620	4/21/71	-90/5	50	-	65	-	62	-	2813.5	196.5	234	
1624	4/21/71	-90.5	50	-	-	-	63	-	2815	198	238	
1627	4/21/71	-90	50	-	-	-	63	59	2816	199	241	
1632	4/21/71	-90	50	-	-	-	62	-	2816.5	199.5	246	
1636	4/21/71	-90	50	-	65	0.2	62	58	2817	200	250	
1643	4/21/71	-90	50	-	-	-	60	57	2818	201	257	
1646	4/21/71	-90	51	79	-	-	60	-	2818	201	260	
1650	4/21/71	-90	51	-	65	0.2	58	57	2818.5	201.5*	264	Closed B_2 feed.
1920	4/21/71	-89.5	55	78	65	0.1	-	52	2818	-	-	
2340	4/21/71	-89	55	78	65	0	-	48	2817	-	-	
0825	4/22/71	-87	58	78	65	0	-	50	2815	-	-	
0835	4/22/71	-	-	-	-	-	-	-	2803.5	203.5*	-	Weight w/o dry ice
1115	4/22/71	-86	29	-	-	0.3	-	49	2809.5	-	-	Started topping @0845
1200	4/22/71	-86	26	-	-	0.3	-	49	2808	-	-	Stopped topping
1242	4/22/71	-	22	-	-	-	-	-	2811	-	-	Started topping
1310	4/22/71	-86	22	-	-	0.3	-	49	2807	-	-	
1330	4/22/71	-	20	-	-	-	-	-	2806.5	-	-	
1345	4/22/71	-86	19	-	-	-	-	55	2805.5	-	-	Gas Sample 2
1350	4/22/71	-86	18	-	-	-	-	-	2805.5	-	-	Finished topping
1400	4/22/71	-	-	-	-	-	-	-	2796.5	196.5	-	Weight w/o dry ice
1403	4/22/71	-86	18	-	-	-	-	-	2805	197	264	Weight with dry ice
1405	4/22/71	-	19	-	-	-	105	-	2805.5	197.5	266	Restarted B_2 feed
1406	4/22/71	-	20	-	-	-	-	-	2806	198	267	
1407	4/22/71	-	-	-	-	-	-	-	2806	198	268	Increased feed rate
1408	4/22/71	-	20	-	-	-	-	-	2807	199	269	
1409	4/22/71	-	20	-	-	-	-	-	2808	200	270	
1410	4/22/71	-	-	-	-	0.3	90	49	2809	201	271	
1411	4/22/71	-	21	-	-	-	-	-	2810	202	272	
1411.5	4/22/71	-	21	-	-	-	-	-	2810.5	202.5	272.5	Stopped feeding
1412	4/22/71	-	-	-	-	-	-	-	2811	203	273	Weight with dry ice
1415	4/22/71	-	-	-	-	0.2	110	50	2803	203.0		Weight w/o dry ice
1525	4/22/71	-86	18	-	-	0.3	-	51	2810.75	203.0	-	Weight with dry ice

*Actual B_2H_6 weight 203.5 due to 2.0 lbs. dry ice loss during charging.

TABLE 9 (PAGE 5 of 5)

DIBORANE SHIPPING CONTAINER
DIBORANE CHARGE FOR 30-DAY STORAGE TEST

TIME	DATE	TI °C	PI PSIG	VI %	LI %	VAC	COND INLET PSIG	AMB TEMP °F	SCALE READING LBS	B_2H_6 WEIGHT LBS	CHARGE TIME MIN.	REMARKS
1540	4/22/71	-	-	78	66	-	-	-	2810.5	203.5	-	Start topping
1545	4/22/71	-86	19	-	-	0.3	-	52	2810	-	-	
1555	4/22/71	-86	18	-	-	-	-	-	2810	-		Gas Sample 3
1600	4/22/71	-86.5	18	78	65	0.3	-	53	2809.5	-	-	Finished topping
1601	4/22/71	-	-	-	-	-	-	-	2801.5	201.5	-	Weight w/o dry ice
1615	4/22/71	-	-	-	-	-	-	-	2781.5	201.5	-	Disconnected
1617	4/22/71	-	-	-	-	-	-	-	2783	-	-	PSV Connected
1645	4/22/71	-85.5	18	78	0	0.3	-	54	2891	-	-	Dry ice charged
												Start of test

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST DATA

TIME	DATE	DAYS	CONTAINER TI °C	CONTAINER PI PSIG	CONTAINER VI* %	CONTAINER LI* %	CONTAINER VACUUM MICRONS	AMB TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
1645	4/22/71	0.00	-85.5	18	78	0	0.3	-	2891.0	108	Start of test
1700	4/22/71	0.01	-82	18	-	-	0.9	74	2891.0	108	
1915	4/22/71	0.10	-82	19	-	-	1.4	69	2890.75	107.75	
2145	4/22/71	0.21	-82	19.5	-	-	1.8	73	2890.0	107	
2400	4/22/71	0.30	-82	19.5	-	-	1.8	71	2889.25	106.25	
0810	4/23/71	0.64	-82	20.5	79	0	2.0	72	2887.0	104	
0840	4/23/71	0.66	-82	20.5	-	-	2.0	71	2887.0	104	
1505	4/23/71	0.93	-82	21	-	-	2.0	72	2885.0	102	
1645	4/23/71	1.00	-82	21	81	0	2.1	76	2884.5	101.5	
1915	4/23/71	1.10	-82	22	-	-	2.3	77	2883.75	100.75	
0045	4/24/71	1.33	-82	23	82	0	2.4	78	2881.75	98.75	
0815	4/24/71	1.65	-82	23	82	0	2.3	74	2879.75	96.75	
1755	4/24/71	2.05	-82	24	82	0	2.3	72	2876.25	93.25	
0815	4/25/71	2.65	-82	26	82	0	2.3	73	2872.0	89	
1330	4/25/71	2.86	-82	26	82	0	2.6	74	2870.0	87	
2400	4/25/71	3.30	-82	27	82	0	2.7	77	2866.0	83	
0815	4/26/71	3.65	-82	27.5	81	0	2.5	82	2863.5	80.5	
0845	4/26/71	3.67	-82	27.5	82	0	2.7	73	2863.25	80.25	
1525	4/26/71	3.94	-82	28	-	-	2.7	72	2861.5	78.5	
2345	4/26/71	4.29	-81	28	82	0	2.9	74	2857.75	74.75	
0810	4/27/71	4.64	-81	29	82	0	2.8	74	2855.0	72	
0840	4/27/71	4.66	-81	29	82	65	2.9	73	2854.5	71.5	
1305	4/27/71	4.85	-81	29	82	65	3.0	72	2852.75	69.75	
1600	4/27/71	4.97	-81	29	-	-	3.0	76	2852.0	69	
1645	4/27/71	5.00	-80.5	29.5	81	65	2.9	73	2851.75	68.75	
1930	4/27/71	5.11	-80.5	29.5	81	65	3.1	77	2850.75	67.75	
2400	4/27/71	5.30	-80.5	29.5	81	65	3.3	75	2849.0	66.0	
0810	4/28/71	5.64	-80.5	30	81	65	3.0	72	2846.0	63.0	
0845	4/28/71	5.67	-80	30	81	65	3.2	74	2846.0	63.0	
1500	4/28/71	5.93	-80	30.5	81	65	3.8	77	2844.0	61.0	
1930	4/28/71	6.11	-80	31	81	65	3.4	75	2842.0	59.0	

*LI = Level Indicator; VI = Voltage Indicator

TABLE 10 PAGE 2 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST DATA

TIME	DATE	DAYS	CONTAINER TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
2345	4/28/71	6.29	-80	31	81	65	3.4	74	2840.75	57.75	
0810	4/29/71	6.64	-80	31	81	65	3.5	76	2837.5	54.5	
0845	4/29/71	6.67	-80	31	81	0	3.7	75	2837.75	54.75	
1320	4/29/71	6.86	-79.5	31	82	0	3.7	76	2835.75	52.75	
1500	4/29/71	6.93	-80	31	-	-	3.5	78	2835.5	52.5	
1615	4/29/71	6.98	-79	32	82	0	3.8	78	2835.0	52.0	
1915	4/29/71	7.10	-79	32	82	0	3.7	75	2834.0	51.0	
2350	4/29/71	7.30	-79	32	82	0	3.7	75	2832.25	49.25	
0810	4/30/71	7.64	-79	32	81	65	3.8	74	2829.5	46.5	
0840	4/30/71	7.66	-79	33	81	65	3.9	75	2829.25	46.25	
1555	4/30/71	7.97	-79	32	-	-	3.8	72	2827	44.0	
1600	4/30/71	7.97	-79	33	81	65	4.0	77	2826.75	43.75	
1915	4/30/71	8.10	-79	33	81	65	3.8	74	2825.75	42.75	
2345	4/30/71	8.29	-79	33.5	81	65	3.8	73	2824	41.0	
0910	5/1/71	8.68	-78.5	34	81	65	4.0	76	2821	38.0	
1745	5/1/71	9.04	-78.5	34.5	81	65	3.9	74	2818	35.0	
0820	5/2/71	9.65	-78	35	81	65	4.0	75	2812.75	29.75	
1325	5/2/71	9.86	-78	35	81	65	4.0	74	2811.0	28.0	
1915	5/2/71	10.10	-78	35	81	65	3.9	73	2808.75	25.75	
2345	5/2/71	10.29	-78	35	81	65	4.0	74	2807.25	24.25	
0810	5/3/71	10.64	-78	35	81	65	4.0	75	2804.0	21.0	
0845	5/3/71	10.67	-78	36	81	65	4.2	77	2804.0	21.0	
1550	5/3/71	10.96	-78	36	-	-	4.0	78	2801.5	18.5	
1645	5/3/71	11.00	-77.5	36	81	65	4.1	75	2801.25	18.25	
1930	5/3/71	11.11	-77.5	36	81	65	3.9	75	2800.25	17.25	
2345	5/3/71	11.29	-77.5	36	81	65	3.9	76	2798.75	15.75	
0810	5/4/71	11.64	-77.5	37	81	65	3.9	71	2796.0	13.0	
0900	5/4/71	11.68	-77.5	37	81	65	4.2	75	2796.0	13.0	

TABLE 10 PAGE 3 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST DATA

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
1340	5/4/71	11.92	-77	37	81	65	4.2	73	2794.5	11.5	
1520	5/4/71	11.94	-77	38	-	-	4.0	72	2794	11.0	
1915	5/4/71	12.10	-77	37	81	65	4.2	78	2793	10.0	
2345	5/4/71	12.29	-76	38	81	65	4.3	75	2791.5	8.5	
0810	5/5/71	12.64	-76	39	81	65	4.2	72	2789.0	6.0	
0900	5/5/71	12.68	-75	39	81	66	4.4	74	2788.0	5.0	
1420	5/5/71	12.90	-74.5	39.5	80	67	5.1	78	2787.5	4.5	
1525	5/5/71	12.94	-75	40	-	-	4.8	75	2787.0	4.0	
1700	5/5/71	13.01	-73.5	40	81	67	4.8	75	2786.75	3.75	
1915	5/5/71	13.10	-73.5	40	81	67	4.8	78	2786	3.0	
2345	5/5/71	13.29	-72.5	41	81	67	4.8	75	2785	2.0	
0810	5/6/71	13.64	-71	42	81	67	4.9	76	2783	0	
0855	5/6/71	13.67	-71	42	81	67	5.0	78	2783.25	0.25	
1350	5/6/71	13.88	-68	43	80	67.5	5.1	74	2782.25	-0.25	
1545	5/6/71	13.96	-66	43	-	-	4.9	72	2782.5	-0.5	
1710	5/6/71	14.02	-61	46	81	68.5	5.1	75	2782.25	-0.75	
1915	5/6/71	14.10	-58.5	47	81	69	5.1	75	2782.5	-0.5	
2400	5/6/71	14.30	-54	49	81	69	5.6	75	2782.25	-0.75	
0810	5/7/71	14.64	-51	52	81	71	5.5	74	2782.25	-0.75	
0840	5/7/71	14.66	-50	53.5	81	71	5.7	75	2782.5	-0.5	
1305	5/7/71	14.85	-49.5	58	81	71	6.1	76	2782.5	-0.5	
1530	5/7/71	14.95	-49	59	-	-	6.2	78	2782.25	-0.75	
1645	5/7/71	15.00	-48	60	81	72	6.7	79	2782.5	-0.5	
1915	5/7/71	15.10	-47	61	81	72	6.6	76	2782.5	-0.5	
2345	5/7/71	15.29	-46.5	63	81	71	6.3	75	2782.5	-0.5	
1210	5/8/71	15.81	-44	72	81	71	6.6	74	2782.75	-0.25	
1755	5/8/71	16.05	-43	74	81	70	6.7	75	2782.5	-0.5	
0830	5/9/71	16.66	-41	83	81	70	6.8	74	2782.5	-0.5	
1340	5/9/71	16.87	-40.5	88	81	71	7.7	78	2782.25	-0.75	
1745	5/9/71	17.04	-40	90	81	72	8.3	78	2782.5	-0.5	

TABLE 10 PAGE 4 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST DATA

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
2030	5/9/71	17.16	-39.5	91	81	71	8.1	76	2782.5	-0.5	
2400	5/9/71	17.30	-39.5	93	81	71	7.9	76	2782.25	-0.75	
0815	5/10/71	17.65	-39.5	100	81	71	7.5	82	2782.25	-0.75	
0820	5/10/71	17.65	-39	100	-	-	7.5	-	-	-	
1320	5/10/71	17.86	-38.5	102	81	72	8.8	80	2782.5	-0.5	
1550	5/10/71	17.96	-39	102	-	-	9.0	80	2782.25	-0.75	
1600	5/10/71	17.97	-37.5	103	81	71	9.4	81	2782.5	-0.5	
1915	5/10/71	18.10	-37	106	81	71.5	9.7	80	2782.5	-0.5	
2145	5/10/71	18.21	-36	108	81	71	9.1	75	2782.5	-0.5	
2345	5/10/71	18.29	-36	109	81	71	9.1	75	2782.5	-0.5	
0810	5/11/71	18.64	-35	112	81	71	8.5	72	2782.25	-0.75	
0840	5/11/71	18.66	-35	113	81	70	8.8	76	2782.25	-0.5	
1105	5/11/71	18.76	-35	117	81	71	9.6	80	2782.5	-0.5	
1320	5/11/71	18.86	-34.5	118	81	71	10.1	81	2782.5	-0.5	
1500	5/11/71	18.93	-34	119	81	72	11.0	82	2782.5	-0.5	
1535	5/11/71	18.95	-34	119	-	-	10.5	83	2782.25	-0.75	
1645	5/11/71	19.00	-33.5	120	81	71	11.5	84	2782.5	-0.5	
1725	5/11/71	19.03	-33	120	81	71	11.5	84	2782.5	-0.5	
1915	5/11/71	19.10	-32	121	63	86	11.5	81	2782.5	-0.5	Alarm Sounded
0100	5/12/71	19.34	-31	127	55	100	10.5	76	2782.5	-0.5	
0815	5/12/71	19.65	-30.5	130	21	30	10.0	74	2782.5	-0.5	
0830	5/12/71	19.66	-30	131	55	100	10.2	75	2782.5	-0.5	
1315	5/12/71	19.85	-30	133	13	0	11.0	79	2782.5	-0.5	
1600	5/12/71	19.97	-30	136	-	-	10.5	74	2782.5	-0.5	
1715	5/12/71	20.02	-29.5	137	22	28	10.5	74	2782.5	-0.5	
1915	5/12/71	20.10	-29.5	138	24	29	10.2	75	2782.5	-0.5	
2345	5/12/71	20.29	-29.5	140	23	32	10.2	74	2782.5	-0.5	
0810	5/13/71	20.64	-29	148	24	40	9.9	72	2782.5	-0.5	
0835	5/13/71	20.66	-29	147	24	32	10.2	75	2782.5	-0.5	
1645	5/13/71	21.00	-28	152	25	30	9.8	73	2782.5	-0.5	

TABLE 10 PAGE 5 OF 5

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST DATA

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
1905	5/13/71	21.10	-28	153	21	28	9.8	75	2782.5	-0.5	
0140	5/14/71	21.37	-27.5	158	25	32	9.8	74	2782.5	-0.5	
0810	5/14/71	21.64	-26.5	160	24	39	9.8	72	2782.5	-0.5	
0845	5/14/71	21.67	-26.5	163	-	-	9.7	74	2782.5	-0.5	
1340	5/14/71	21.87	-26	166	-	-	10.5	77	2782.5	-0.5	Start Sloshing
1405	5/14/71	21.89	-21.5	166	-	-	11.0	77	2782.0	-1.0	Finish Sloshing
1915	5/14/71	22.10	-21.5	170	-	-	11.5	76	2782.5	-0.5	
0100	5/15/71	22.34	-21.5	173	-	-	11.0	80	2782.5	-0.5	
0945	5/15/71	22.71	-21.0	180	-	-	10.5	75	2782.5	-0.5	
1745	5/15/71	23.04	-20.5	187	-	-	13.0	82	2782.75	-0.25	
2200	5/15/71	23.22	-20.0	190	-	-	13.0	79	2782.75	-0.25	
0830	5/16/71	23.66	-19.5	197	-	-	12.5	75	2782.75	-0.25	
1400	5/16/71	23.89	-19.5	202	-	-	15.0	84	2782.75	-0.25	
0815	5/17/71	24.65	-18	215	25	30	12.5	72	2782.5	-0.5	
1600	5/17/71	24.97	-17	220	-	-	16	84	2782.5	-0.5	
1730	5/17/71	25.03	-18	220	-	-	17	83	-	-	
0800	5/18/71	25.64	-14	231	-	-	13	70	-	-	
0810	5/18/71	25.64	-15	232	24	30	14	72	2782.5	-0.5	
1550	5/18/71	25.96	-14	240	-	-	19	92	2782.5	-0.5	
0800	5/19/71	26.64	-11	251	-	-	16	74	-	-	
0807	5/19/71	26.64	-10.5	250	0	0	16	74	2782.5	-0.5	
1545	5/19/71	26.96	-10	260	-	-	21	91	2782.5	-0.5	
0805	5/20/71	27.64	-10	270	3	0	19	76	2782.5	-0.5	
1545	5/20/71	27.96	- 9.9	280	-	-	21	85	2782.5	-0.5	
0807	5/21/71	28.64	- 9.5	290	3	0	17	72	2782.5	-0.5	
0815	5/21/71	28.65	- 8	290	-	-	17.5	75	-	-	
1500	5/21/71	28.93	- 9	300	-	-	20	82	2782.5	-0.5	End of Test
1520	5/21/71	28.94	-10	290	-	-	19.5	80	2848.0	65.5	Dry Ice Added

TABLE 11

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST RESULTS
SMOOTHED DATA-LINEAR SEGMENTS

TIME DAYS	DRY ICE LBS	PRES. PSIG	TEMP °C. ACTUAL	TEMP °C. INDIC.	REMARKS
0.00	108.0	18	-79.0	-85.5	Start
0.01	-	-	-	-82	
0.10	-	19	-	-	
0.21	-	-	-77.5	-	
1.65	96.75	-	-	-	
2.65	-	26	-73.0	-	
11.29	15.75	-	-	-	
11.64	-	-	-	-77.5	
12.29	8.5	-	-	-	
12.68	-	39	-	-	
13.10	-	-	-65.8	-	
13.64	-	-	-	-71	
13.67	-	42	-65.0	-	
13.88	0	-	-	-68	
14.02	-	-	-	-61	
14.30	-	49	-62.1	-54	
15.10	-	-	-	-47	
15.81	-	-	-53.8	-	
16.66	-	-	-	-41	
17.86	-	-	-	-38.5	
18.29	-	-	-43.0	-	
19.03	-	120	-	-	
19.10	-	-	-	-32	Alarm
19.66	-	-	-	-30	
20.66	-	-	-34.7	-	
21.87	-	-	-	-26	
21.89	-	-	-	-21.5	Slosh
22.34	-	173	-	-	
23.22	-	-	-27.0	-	
24.65	-	-	-	-18	
25.64	-	231	-	-	
26.64	-	-	-17.6	-	
26.96	-	-	-	-10	
28.93	0	300	-11.0	- 9	
28.94	65.5	290	-12.3	-10	Dry Ice Added
29.72	22.5	140	-36.0	-33	
29.98	13.5	125	-39.2	-39	

TABLE 12

DIBORANE SHIPPING CONTAINER
30-DAY STORAGE TEST RESULTS
SMOOTHED DATA-EVEN DAYS

TIME, DAYS	DRY ICE, POUNDS	PRESSURE PSIG	TEMP., °C
0	108.0	18.0	-79.0
1	101.2	21.5	-76.0
2	93.8	24.2	-74.2
3	85.4	26.5	-72.8
4	77.0	27.7	-72.1
5	68.6	29.0	-71.4
6	60.2	30.3	-70.7
7	51.8	31.6	-70.0
8	43.4	32.9	-69.3
9	35.0	34.2	-68.6
10	26.6	35.5	-67.9
11	18.2	36.8	-67.2
12	10.6	38.1	-66.6
13	4.7	40.0	-65.9
14	0	45.7	-63.5
15	0	59.5	-58.3
16	0	74.5	-53.0
17	0	89.5	-48.6
18	0	104.5	-44.3
19	0	119.5	-40.5
20	0	135.5	-37.0
21	0	151.5	-33.7
22	0	167.6	-30.7
23	0	184.6	-27.7
24	0	202.2	-24.9
25	0	219.8	-22.1
26	0	238.6	-19.4
27	0	259.5	-16.6
28	0	280.5	-13.7
*29	0	301.5	-10.8
*30	0	322.4	- 7.9
*31	0	343.4	- 5.3
*32	0	364.4	- 2.8
*33	0	385.4	- 0.5
*34	0	406.3	+ 1.6

*Extrapolation

TABLE 13 PAGE 1 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	VI* %	LI* %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
0807	5/21/71	28.64	- 9.5	290	3	0	17	72	2782.5	-0.5	
0815	5/21/71	28.65	- 8	290	-	-	17.5	75	-	-	
1500	5/21/71	28.93	- 9	300	-	-	20	82	2782.5	-0.5	End of Test
1520	5/21/71	28.94	-10	290	-	-	19.5	80	2848.0	65.5	Dry Ice Added
1000	5/22/71	29.72	-33	140	-	-	10.5	70	2805.0	22.5	
1620	5/22/71	29.98	-39	125	-	-	10.0	74	2796.0	13.5	
1630	5/22/71	29.99	-41	121	-	-	9.5	74	2866.0	83.5	
2245	5/23/71	31.25	-53	70	-	-	6.0	68	2830.5	48.0	
0815	5/24/71	31.65	-59	60	5	0	4.9	68	2822.5	40.0	
1600	5/24/71	31.97	-60	57	-	-	5.9	80	2816.5	34.0	
0805	5/25/71	32.64	-62	50	5	0	5.3	76	2805.0	22.5	
1550	5/25/71	32.96	-65	48	-	-	5.5	78	2800.0	17.5	
0010	5/26/71	33.31	-63.5	43	-	-	5.0	74	2796.0	13.5	
0807	5/26/71	33.64	-66	42	5	0	4.1	69	2792.5	10.0	
1500	5/26/71	33.93	-65	40	-	-	4.3	68	2790.0	7.5	
2200	5/26/71	34.22	-66	42	-	-	4.3	71	2787.5	5.0	
0810	5/27/71	34.64	-66	42	5	0	3.9	66	2785.0	2.5	
1535	5/27/71	34.95	-66	45	-	-	4.5	71	2783.5	1.0	
0930	5/28/71	35.70	-55	52	-	-	4.0	63	2782.5	0	
1500	5/28/71	35.93	-52	59	-	-	5.2	72	2782.5	0	
1545	5/28/71	35.96	-60	55	-	-	4.8	68	2867.5	85.0	Dry Ice Added
2215	5/28/71	36.24	-63	47	-	-	3.4	67	2860.5	78.0	
1030	5/29/71	36.74	-68	42	-	-	2.6	65	2852.5	70.0	
1750	5/30/71	38.04	-71	37	-	-	3.8	76	2837.0	54.5	
1140	5/31/71	38.79	-72	36	-	-	4.2	74	2829.0	46.5	
2400	5/31/71	39.30	-72	34	-	-	3.8	75	2824.0	41.5	
0810	6/1/71	39.64	-72	35	5	0	3.6	72	2820.5	38.0	
1555	6/1/71	39.97	-72	35	-	-	5.5	88	2817.0	34.5	
2050	6/1/71	40.17	-72	32	-	-	5.4	83	2815.0	32.5	
0815	6/2/71	40.65	-72	32	5	0	4.9	78	2810.0	27.5	
1600	6/2/71	40.97	-72	31	-	-	4.9	77	2807.5	25.0	
0810	6/3/71	41.64	-72	31	5	0	4.9	77	2801.5	19.0	

* LI = Level Indicator; VI = Voltage Indicator

TABLE 13 PAGE 2 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
1600	6/3/71	41.97	-72	31	-	-	6.0	88	2798.5	16	
0810	6/4/71	42.64	-72	31	-	-	4.9	76	2792.5	10	
1405	6/4/71	42.89	-72	31	-	-	7.0	90	2790.5	8	
1425	6/4/71	42.90	-72	31	-	-	6.9	86	2853.5	71	Dry Ice Added
1925	6/4/71	43.11	-73	33	-	-	5.4	83	2850	67.5	
1145	6/5/71	43.79	-74	31	-	-	4.4	80	2842	59.5	
1430	6/6/71	44.91	-74	31	-	-	4.7	83	2833	50.5	
2345	6/6/71	45.29	-74	30	-	-	3.2	70	2826.5	44	
0815	6/7/71	45.65	-74	30	-	-	3.1	70	2823.5	41	
1530	6/7/71	45.95	-74	30	-	-	5.5	82	2821.5	39	
2400	6/7/71	46.30	-76	29	-	-	5.1	82	2817	34.5	
0810	6/8/71	46.64	-77	30	-	-	4.8	80	2813.5	31	
1600	6/8/71	46.97	-77	30	-	-	5.9	86	2810.5	28	
2100	6/8/71	47.18	-77	29	-	-	4.8	79	2809	26.5	
0815	6/9/71	47.65	-77	30	-	-	4.1	72	2804.5	22	
1600	6/9/71	47.97	-77	30	-	-	5.5	82	2801.5	19	
2400	6/9/71	48.30	-77	31	-	-	4.6	75	2799	16.5	
0810	6/10/71	48.64	-77	31	-	-	4.0	75	2795.5	13	
1605	6/10/71	48.97	-55	35	-	-	6.0	71	2790.5	8	TI Maintenance
2345	6/10/71	49.29	-55	32	-	-	5.1	77	2788	5.5	
0810	6/11/71	49.64	-55	32	-	-	4.5	70	2786	3.5	
1000	6/11/71	49.72	-55	32	-	-	5.0	76	2785.5	3	
1020	6/11/71	49.73	-55	32	-	-	4.5	76	2885.5	103	
1120	6/11/71	49.77	-58	32	-	-	-	-	-	-	
1310	6/11/71	49.85	-56	32	-	-	-	-	-	-	Meas. Cntr. Grav
1355	6/11/71	49.85	-55	33	-	-	-	-	-	-	
1540	6/11/71	49.96	-58	31	-	-	6.0	87	2884	101.5	
1945	6/11/71	50.12	-58	33	-	-	6.2	86	2881	98.5	
1445	6/12/71	50.92	-58	31	-	-	5.5	83	2870	87.5	
1705	6/13/71	52.01	-58	31	-	-	6.1	90	2856	73.5	

TABLE 13 PAGE 3 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP. °F	SCALE READING LBS.	DRY ICE, LBS.	REMARKS
2400	6/13/71	52.30	-58	29	-	-	4.9	80	2852	69.5	
0810	6/14/71	52.64	-58	29	-	-	4.7	78	2848.5	66	
1550	6/14/71	52.96	-58	29	-	-	5.2	86	2845	62.5	
2400	6/14/71	53.30	-57	28	-	-	5.1	79	2841	58.5	
0810	6/15/71	53.64	-58	29	-	-	4.7	78	2837	54.5	
1600	6/15/71	53.97	-58	29	-	-	5.0	82	2833.5	51	
2400	6/15/71	54.30	-58	28	-	-	4.8	77	2829.5	47	
0810	6/16/71	54.64	-58	29	-	-	4.5	76	2826.5	44	
1555	6/16/71	54.97	-58	29	-	-	5.3	84	2823.5	41	
2400	6/16/71	55.30	-58	30	-	-	5.1	77	2819	36.5	
0810	6/17/71	55.64	-58	30	-	-	4.0	71	2815.5	33	
1550	6/17/71	55.96	-58	30	-	-	5.5	81	2812.5	30	
2400	6/17/71	56.30	-58	31	-	-	5.2	79	2809	26.5	
0810	6/18/71	56.64	-58	31	-	-	4.5	74	2806	23.5	
1605	6/18/71	56.97	-58	32	-	-	7.0	92	2803	20.5	
2400	6/18/71	57.30	-58	32	-	-	6.1	83	2799.5	17	
1650	6/19/71	58.00	-58	33	-	-	7.7	95	2793	10.5	
1645	6/20/71	59.00	-53	37	-	-	9.0	96	2785.5	3	
2345	6/20/71	59.29	-48	38	-	-	9.1	88	2784	1.5	
0815	6/21/71	59.65	-38	42	-	-	10	82	2783.5	1	
1520	6/21/71	59.94	-30	49	-	-	11	86	2783.5	1	
1555	6/21/71	59.97	-30	49	-	-	11	86	2848.5	66	
2400	6/21/71	60.30	-48	42	-	-	7.7	81	2840	57.5	
0810	6/22/71	60.64	-50	39	-	-	6.5	76	2835	52.5	
1550	6/22/71	60.96	-50	39	-	-	7.9	90	2830.5	48	
2345	6/22/71	61.29	-49	36	-	-	6.8	82	2826	43.5	
0815	6/23/71	61.65	-50	35	-	-	5.8	74	2821	38.5	
0900	6/23/71	61.68	-	-	-	-	-	-	2837.5	38.5	
0910	6/23/71	61.68	-50	35	-	-	5.8	76	2837.5	38.5	Sampled
1300	6/23/71	61.85	-	42	-	-	-	-	2830	36	Rmv. 5.5# B_2

TABLE 13 PAGE 4 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
1555	6/23/71	61.97	-51	42	-	-	7.4	92	2827.5	33.5	
2400	6/23/71	62.30	-52	41	-	-	6.3	82	2823	29	
0810	6/24/71	63.64	-52	41	-	-	5.5	78	2820	26	
1600	6/24/71	63.97	-52	41	-	-	7.9	95	2816	22	
2345	6/24/71	64.29	-52	40	-	-	6.7	86	2812	18	
0810	6/25/71	64.64	-52	41	-	-	6.9	80	2809	15	
1515	6/25/71	64.94	-52	41	-	-	6.9	91	2875	81	
1745	6/26/71	66.04	-53	38	-	-	7.1	93	2857.5	63.5	
1745	6/27/71	67.04	-54	38	-	-	8.1	97	2845	-	
2400	6/27/71	67.30	-54	39	-	-	6.8	88	2842	48	
0810	6/28/71	67.64	-54	39	-	-	6.2	86	2838.5	44.5	
1555	6/28/71	67.97	-54	39	-	-	8.5	102	2829	40	Rmv. 4# B_2
0815	6/29/71	68.65	-54	39	-	-	6.5	88	2821	32	
1600	6/29/71	68.97	-54	39	-	-	8.6	103	2811	27	Rmv. 5# B_2
2345	6/29/71	69.29	-53	38	-	-	7.7	92	2808	24	
2345	6/30/71	70.29	-53	39	-	-	7.7	93	2797	13	
0810	7/1/71	70.64	-53	40	-	-	7.0	90	2793	9	
1600	7/1/71	70.97	-53	40	-	-	7.6	91	2790	6	
2345	7/1/71	71.29	-53	40	-	-	7.2	86	2787.5	3.5	
0815	7/2/71	71.65	-53	41	-	-	7.0	82	2785	1	
1515	7/2/71	71.94	-50	45	-	-	9.1	86	2874	90	
2345	7/2/71	72.29	-51	42	-	-	5.7	76	2867	83	
1400	7/3/71	72.89	-51	40	-	-	5.8	80	2859	75	
1715	7/4/71	74.02	-52	39	-	-	6.2	84	2845	61	
1600	7/5/71	74.97	-52	39	-	-	6.3	85	2835	51	
2345	7/5/71	75.29	-52	39	-	-	5.7	80	2832	48	
0810	7/6/71	75.64	-52	39	-	-	5.2	76	2828.5	44.5	
1555	7/6/71	75.97	-52	54	-	-	7.1	86	2782.5	40.5	Rmv. 42# B_2
2400	7/6/71	76.30	-52	54	-	-	6.5	82	2777	35	
0810	7/7/71	76.64	-52	54	-	-	5.9	74	2774	32	
1525	7/7/71	76.94	-52	50	-	-	8.1	90	2732	28	Rmv. 38# B_2
2400	7/7/71	77.30	-52	52	-	-	6.8	84	2726	22	

TABLE 13 PAGE 5 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS	DRY ICE, LBS.	REMARKS
0810	7/8/71	77.64	-52	50	-	-	6.0	78	2723	19	
1600	7/8/71	77.97	-52	50	-	-	8.8	96	2718	14	
2400	7/8/71	78.30	-52	52	-	-	7.5	88	2713	9	
0810	7/9/71	78.64	-52	52	-	-	8.0	88	2711	7	
1520	7/9/71	78.94	-52	52	-	-	8.5	94	2806	102	
2400	7/9/71	79.30	-52	50	-	-	6.4	84	2799	95	
1630	7/10/71	79.99	-52	51	-	-	8.1	93	2789.5	85.5	
1645	7/11/71	81.00	-53	52	-	-	6.1	82	2778	74	
2400	7/11/71	81.30	-53	52	-	-	6.0	78	2774.5	70.5	
0810	7/12/71	81.64	-53	52	-	-	5.9	75	2770.5	66.5	
1600	7/12/71	81.97	-53	52	-	-	7.1	88	2766.5	62.5	
2400	7/12/71	82.30	-53	52	-	-	6.5	79	2762.5	58.5	
0810	7/13/71	82.64	-53	52	-	-	6.0	76	2759	55	
2345	7/13/71	83.29	-53	53	-	-	7.6	87	2751	47	
2200	7/14/71	84.22	-53	53	-	-	7.7	83	2741	37	
2345	7/15/71	85.29	-52	55	-	-	7.6	82	2730	26	
1515	7/16/71	85.94	-	-	-	-	-	-	2791	87	
2215	7/16/71	86.23	-53	52	-	-	6.8	83	2786	82	
1445	7/17/71	86.92	-53	51	-	-	7.1	87	2777	73	
2230	7/18/71	88.24	-53	52	-	-	6.8	82	2762	58	
2145	7/19/71	89.21	-53	52	-	-	7.0	83	2751	47	
2200	7/20/71	90.22	-53	53	-	-	7.2	81	2740	36	
2215	7/21/71	91.23	-53	54	-	-	7.5	82	2729	25	
1605	7/22/71	91.97	-	-	-	-	-	-	-	-	Rmv. 7.5# B_2
2200	7/22/71	92.22	-53	53	-	-	7.7	85	2711	14.5	
2345	7/23/71	93.29	-53	51	-	-	5.8	78	2769	72.5	
1800	7/24/71	94.08	-53	50	-	-	5.0	75	2759.5	63	
2045	7/25/71	95.17	-54	50	-	-	5.8	79	2746.5	50	
2345	7/26/71	96.29	-54	51	-	-	5.2	75	2733	36.5	
1650	7/27/71	97.00	-54	51	-	-	5.8	79	2727.5	31	
1645	7/28/71	98.00	-54	50	-	-	-	77	2718	21.5	
2330	7/29/71	99.00	-54	50	-	-	5.0	69	2706	9.5	

TABLE 13 PAGE 6 OF 6

DIBORANE SHIPPING CONTAINER
POST-TEST RECOOL

TIME	DATE	DAYS	TI °C	PI PSIG	VI %	LI %	VACUUM MICRONS	AMB. TEMP °F	SCALE READING LBS.	DRY ICE, LBS.	REMARKS
1700	7/30/71	100.01	-57	49	-	-	4.9	74	2764	67.5	-
1610	7/31/71	100.98	-57	48	-	-	5.2	76	2753	56.5	
1700	8/1/71	102.01	-57	48	-	-	5.7	79	2742.5	46	
2350	8/2/71	103.30	-56	48	-	-	5.3	75	2730	33.5	
2345	8/3/71	104.29	-56	48	-	-	4.9	73	2720.5	24	
2400	8/4/71	105.30	-56	49	-	-	4.7	67	2711.5	15	
2140	8/5/71	106.20	-56	49	-	-	5.6	72	2703	6.5	
1500	8/6/71	106.93	-	-	-	-	-	-	2697	0.5	
1510	8/6/71	106.93	-	-	-	-	-	-	2783	86.5	
2140	8/6/71	107.20	-56	49	-	-	5.4	73	2778.5	82	
1725	8/7/71	108.03	-57	48	-	-	6.2	81	2769	72.5	
1700	8/8/71	109.01	-57	48	-	-	6.5	83	2759	62.5	

(Ambient Temperature 72 to 78°F)

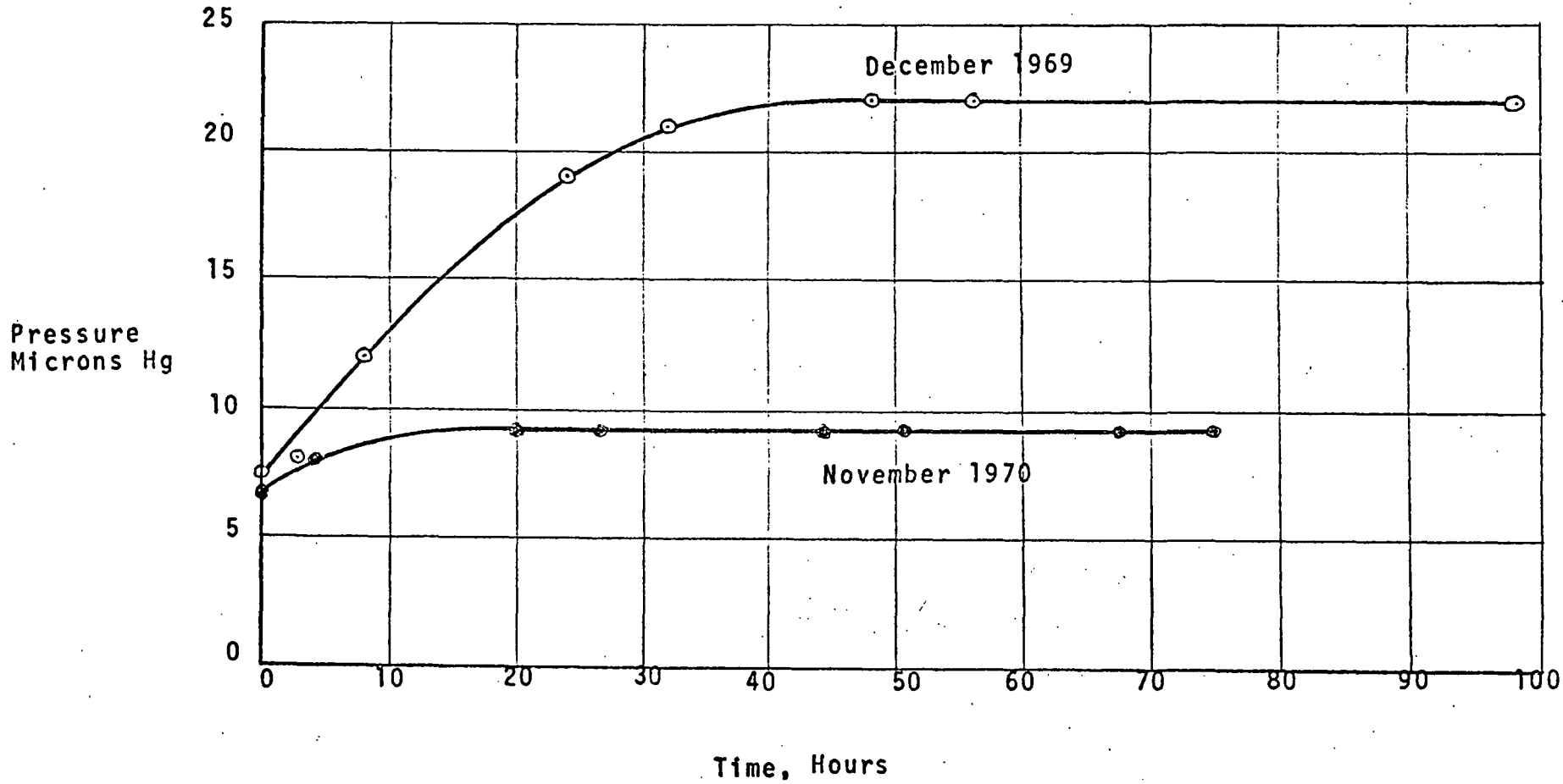


FIGURE 12

VACUUM RETENTION TESTS @ CVI
DIBORANE SHIPPING CONTAINER ANNULAR SPACE

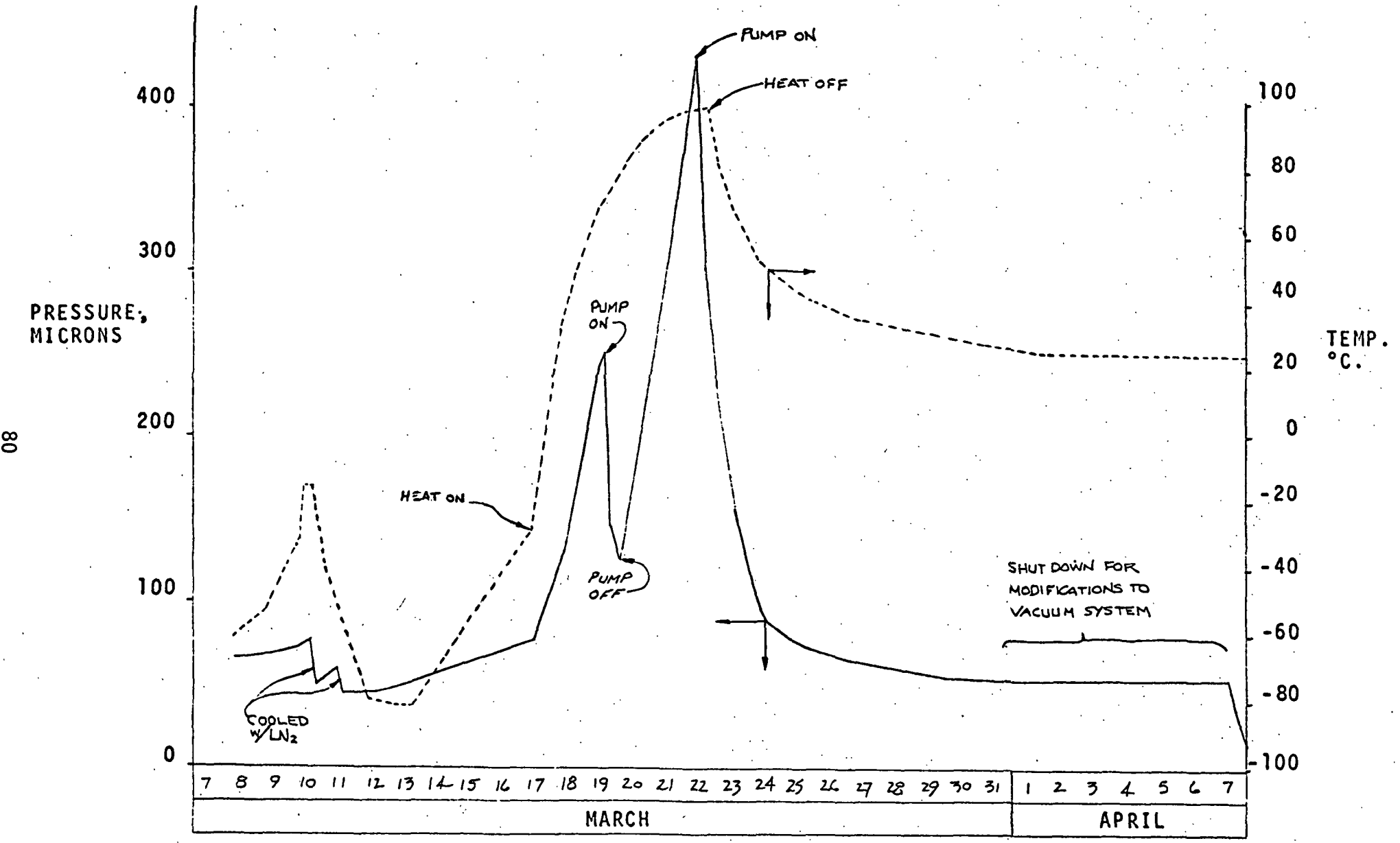


FIGURE 13

REPUMP OF INSULATION SPACE VACUUM

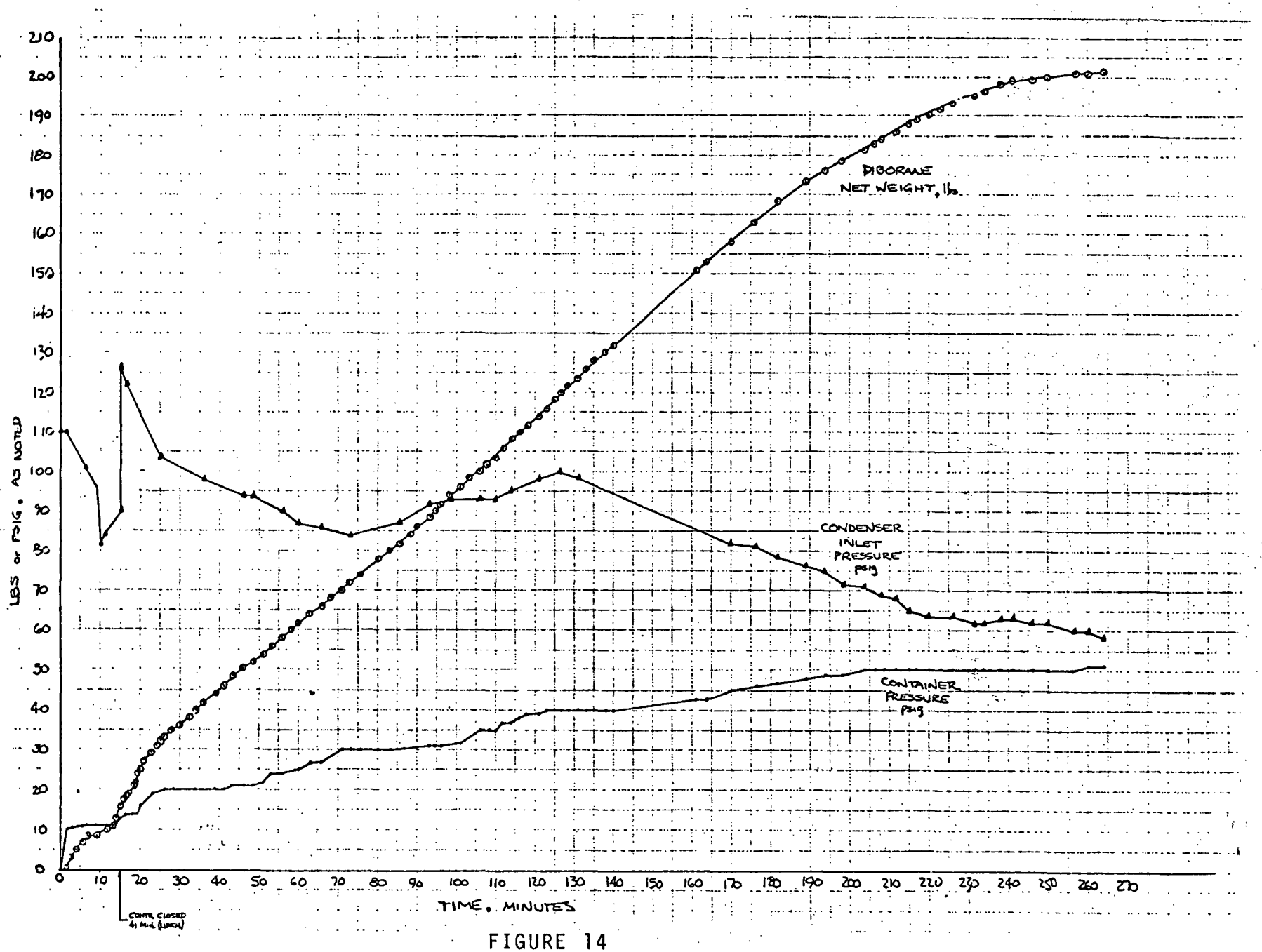


FIGURE 14

DIBORANE CHARGE FOR 30-DAY STORAGE TEST

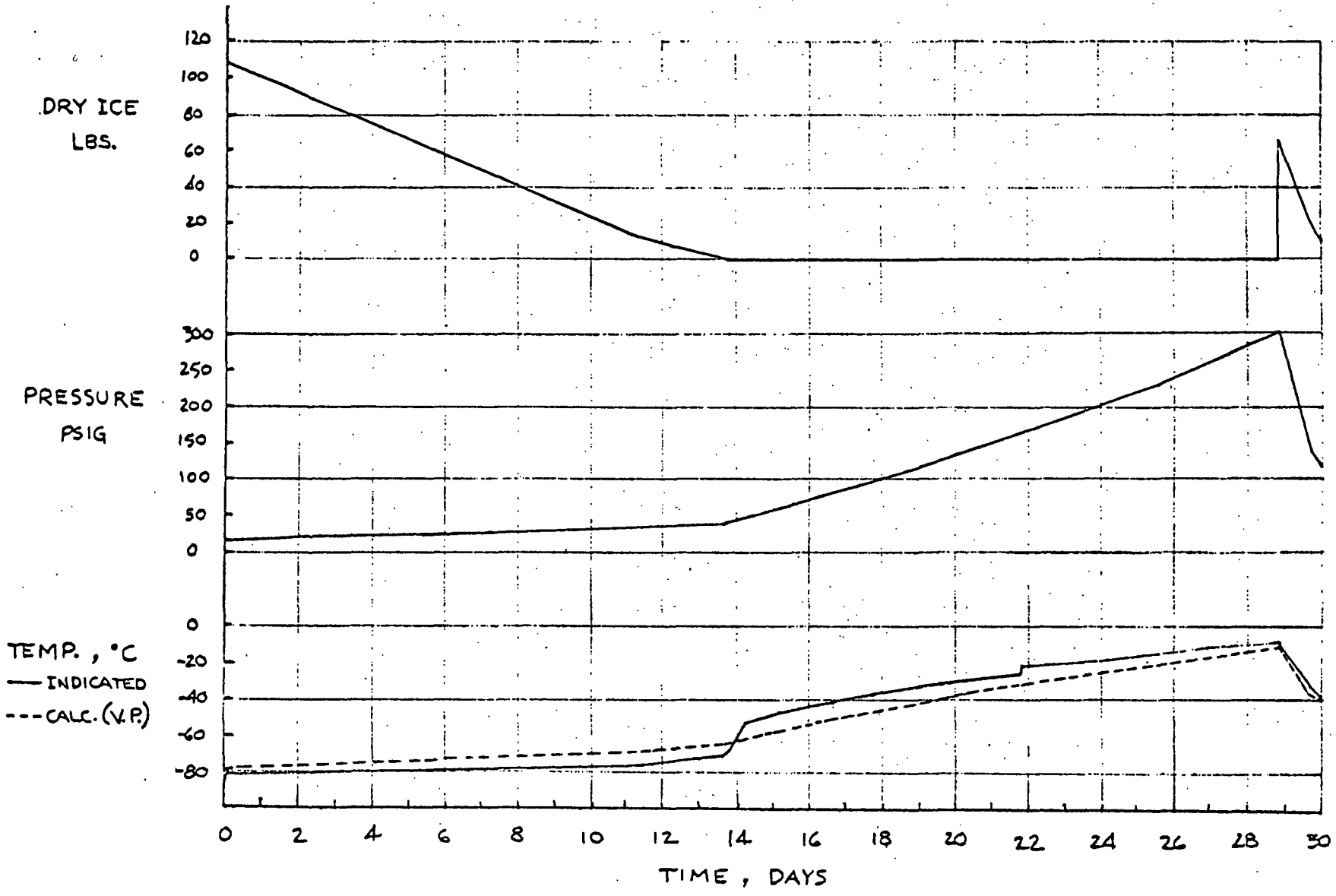


FIGURE 15

30-DAY STORAGE TEST RESULTS

DEPARTMENT OF TRANSPORTATION APPROVAL

PRELIMINARY CONTACTS

Preliminary contacts were made with regulatory agencies concerned with approval of the final container design. In the past it had been advantageous to begin such correspondence during the planning stage of new containers. Accordingly we contacted the U.S. Department of Transportation (Secretary, Hazardous Materials Regulations Board) and Bureau of Explosives, Association of American Railroads (T.C. George, Director and Chief Inspector). In these communications, we outlined the planned project and expressed our intent to periodically submit further information as it was developed.

A letter was received from the Department of Transportation, Office of Hazardous Materials in reply to our letter informing them of the general plans for the diborane shipping container. Their letter indicated lack of background knowledge on shipment of diborane (stating that diborane "has not generally been permitted to be shipped in pure form, but rather in mixtures" and "For small quantities, under temperature controlled conditions, shipment of the pure form has been allowed"). Further, it stated that they "must be very critical in our evaluation because of the very high hazard level product involved".

As in the past when new personnel became involved with approval of diborane shipping permits, it was necessary to visit the DOT office to present the record of our successful diborane shipping and handling. This resulted in a better understanding of diborane shipping, and they expressed a desire to cooperate with us on this project. Since the new container was to have a longer hold time and since it was to use the proven dry ice approach for cooling, there was no reason to anticipate any serious problems in obtaining approval for the larger quantity.

Following the design meeting with CVI on 19 March 1969, contacts were made with representatives of Bureau of Explosives, Association of American Railroads, to report and discuss changes in container design.

DESIGN REVIEW

Upon completion of container design, contacts were made with regulatory agencies to keep them informed for subsequent approval of the final container.

Complete sets of fabrication drawings were provided on 15 July 1969 to Department of Transportation, Hazardous Materials Regulation Board and to Bureau of Explosives, Association of American Railroads. Followup contacts were made regarding coding of the vessel, which was not required for their approval; and there were no objections from these agencies on container design or test plan.

On 8 September 1969 the Bureau of Explosives requested a meeting at their laboratory to discuss the drawings sent to them on 15 July 1969. This meeting was held on 24 September 1969. The functional assembly drawing was helpful to them in understanding the container. A number of questions were answered; and in general they were very well pleased with the container. They felt that tubing leading from the inner vessel was vulnerable to damage with only 16 gage metal around the top. We agreed to explore the possibility of reinforcing this area.

The recommended changes, made to provide additional protection, were reported to the Bureau of Explosives. Revised copies of the functional assembly drawing, showing additional protection for external piping, were sent on 31 October 1969 to Bureau of Explosives, Association of American Railroads and to Department of Transportation, Hazardous Materials Regulation Board.

The Bureau of Explosives replied on 1 December 1969 as follows: "In reply to your letter of October 31 furnishing me drawings of the modifications to the 200 pound diborane vessel, we have now reviewed it and shall take no exceptions."

The Department of Transportation, Office of Hazardous Materials made a belated reply to the information we submitted on 15 July and 3 November 1969. They made favorable comments on the concept and design, and presented some minor questions. These questions were answered in our letter dated 18 December 1969.

REQUEST FOR SPECIAL PERMIT

Contact was reestablished with the Bureau of Explosives, Association of American Railroads, to report having completed testing of the container. They were familiar with this program as a result of our visit there on 24 September 1969, as well as other letter and telephone correspondence. In accordance with the directions received, and as we have done previously for other new containers, the first step toward obtaining a permit was to submit data and as-built drawings to the Bureau of Explosives, as well as to Mr. Gordon Rousseau at Department of Transportation. Mr. Rousseau and other at DOT were also familiar with our container through prior correspondence.

The petition to DOT for special permit is included as Appendix A of this report, since the petition document is a matter of record with DOT and is referred to in the permit.

This written presentation on the the diborane shipping container was sent on 18 June 1971 to Mr. Gordon Rousseau, Chief of Special Permits Branch, Office of Hazardous Materials, Department of Transportation. Copies were also sent to the Bureau of Explosives, Association of American Railroads, as well as to appropriate NASA offices. The written presentation requested a special permit based on a shipping time of fifteen days, and test results were interpreted in support of this request. The container design was also discussed, and complete sets of as-built drawings were provided to DOT and Bureau of Explosives.

DEVELOPMENT OF SPECIAL PERMIT TERMS

Mr. Paul Seay from the DOT Office of Hazardous Materials called Callery on 16 July 1971. Some details of container design and performance were discussed, but primarily the discussion centered around appropriate wording of the special permit. Procedures for safe return of the container were discussed at this time, and again on 21 July 1971.

Net Weight Limits

Callery also recommended a limit on minimum net weight of diborane to be shipped, and a confirming letter was sent to DOT on 29 July 1971 with extrapolated test data for quantities less than 200 pounds. The recommended lower limit was 100 pounds net weight of diborane. This letter was directed to Mr. James Grothe, who replaced Gordon Rousseau as Chief of Special Permits Branch in the DOT Office of Hazardous Materials. Copies were sent to the Bureau of Explosives and NASA offices, as with the initial presentation.

Test Shipment with Methanol

Shipment of methanol in the new container was discussed with Bureau of Explosives, who agreed that it should be permitted; but recommended clearing the shipment with the Department of Transportation. Mr. Raines and Mr. Seay of DOT advised that methanol could not be shipped without a special permit, and Mr. Seay recommended making this part of the diborane permit. Accordingly a confirming letter was sent to DOT on 20 August 1971, with copies to Bureau of Explosives and appropriate NASA offices. The letter recommended a statement to the effect:

> "For container qualification test requirements, methanol (only) may be shipped in this container. Such test with methanol may be repeated as deemed necessary. Quantity range limitations cited for diborane will apply; however, requirement for precooling is not applicable."

Diborane Classification

The DOT Office of Hazardous Materials completed drafting of the special permit on 20 August 1971, and began to circulate it to the various modes of transportation for approval. The permit was approved by the Federal Highway Administration without any questions of Callery. On 13 August 1971 we made a separate contact with the Bureau of Explosives, who said they would take no exceptions.

When the permit reached Federal Railroad Administration, Mr. Quentin Banks of that agency contacted Callery with numerous questions about container design, performance, and procedures. These questions were all answered to his satisfaction; however, during the course of discussions with Mr. Banks, we learned for the first time that DOT-OHM had classified diborane as a Class A Poison. The permit had been written for rail freight shipment, since the Class A Poison classification prohibits rail express shipment.

On 10 September 1971 a letter was sent to Mr. James Grothe at DOT-OHM, with copies to Bureau of Explosives and appropriate NASA offices, requesting they withhold issue of the special permit until the matter of classification had been resolved. The follow-up phone conversations revealed that DOT was proposing a dual classification; that is, the "Class A Poison" is in addition to, rather than instead of, the "Flammable Compressed Gas" label presently used. On 27 October 1971 another letter was sent to Mr. James Grothe at DOT-OHM, with copies to Bureau of Explosives and appropriate NASA offices, outlining information to support our position that diborane is not properly classified as a Class A Poison.

On 9 December 1971 Mr. Grothe reported by telephone that DOT had ruled in favor of a dual classification: "Class A Poison" and "Flammable Compressed Gas". DOT was requested to issue the special permit immediately. A letter was sent 10 December 1971 to confirm that request and to request consideration of approval to ship by rail express in spite of the "Class A Poison" classification, since there are some areas where shipment must be made by rail, and rail freight is too slow for shipment under the limited-time permits. The latter was requested as a modification of the permit, after initial issue to enable start of the methanol shipping test.

Bureau of Explosives

Separate contacts were also made with Bureau of Explosives, who have sole responsibility for approval of the relief system and the refrigeration system. In August the Bureau of Explosives Washington office indicated by telephone that they would take no exceptions. Mr. Clyde Garland of the Washington office called on 13 September 1971 for more detailed information on the relief devices; and this was sent the following day. Then on 18 October 1971 Mr. Charles Schultz from the Bureau of Explosives Raritan Center Laboratory in New Jersey called to request further information on four items: (1) capacity of safety relief devices, (2) burst pressure of the diborane tank, (3) diborane heat of vaporization at 50°F., and (4) heat transfer coefficient of perlite. This information was sent on 20 October 1971 to Mr. Schultz, who called again on 27 October 1971 concerning relief valve capacity. Using an equation from Compressed Gas Association Pamphlet S-1.1 for DOT-4L containers, he calculated a relief valve capacity requirement higher than that of the existing valve. Our reply of 5 November 1971 pointed out that the diborane container does not fit the DOT-4L specification; and calculations were submitted to show that diborane relief capacity available is over three times the capacity needed in a fire situation.

No further questions were received from the Bureau of Explosives.

SPECIAL PERMIT ISSUE AND AMENDMENT

Special Permit Number 6522 was received from DOT on 27 December 1971, and a copy is included at the end of this section of the report. The permit as issued covered only motor vehicle shipment; rail express was still under consideration by DOT.

Department of Transportation, Office of Hazardous Materials failed to act on the request in our 10 december 1971 letter that DOT consider approval of rail express as a mode of transportation for the 200-pound diborane shipping container. In our telephone call on 6 March 1972 to the Federal Railroad Administration, Quentin Banks said rail express probably cannot be given blanket approval on the special permit because of the "Class A Poison" classification for diborane. He did agree, however, that single trip approval would be considered if and when the need arises.

To cover anticipated NASA applications, a letter was sent to DOT-OHM on 3 March 1972 requesting an amendment to DOT Special Permit No. 6522 permitting shipment of quantities less than 100 pounds. Our recommendation was for a graduated shipping time, as follows:

15 days for 100 to 200 pounds
10 days for 50 to 99 pounds
5 days for 6 to 49 pounds

Quantity of 5 pounds or less qualifies as "empty" under the permit, since total decomposition of the diborane would not overpressure the container. Action on this amendment was requested by 15 April 1972.

On 15 March 1972 a letter was sent to DOT recommending the following changes in wording of the permit to prevent misunderstanding which could arise from the language as issued:

Paragraph 6

In line six, the sentence should stop after the word "insulation". The remainder of that sentence should be incorporated in a new sentence: "For diborane shipment, the dry ice chamber must contain not less than 108 pounds of dry ice on the date of shipment." (Or this sentence could be deleted since it is part of paragraph 7).

Insert in line ten (between the sentence ending with "Board." and the sentence beginning with "Except") the sentence previously given in sub-paragraph (7.b.), revised to: "Container must be equipped with an audio-visual alarm to indicate temperature above minus 35°C."

Paragraph 7

Reorganize this "SPECIAL PACKAGING REQUIREMENTS" paragraph to read as follows:

a. All diborane shipments, including those which qualify as "empty" by subparagraph (c), must comply with the following:

i. In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows: "IF NOT DELIVERED BEFORE_____CARRIER MUST ADVISE THE CALLERY CHEMICAL COMPANY, CALLERY, PENNSYLVANIA, ALSO THE BUREAU OF EXPLOSIVES, WASHINGTON, D.C., BY WIRE." The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date of shipment is offered for transportation.

ii. Container must have not less than 108 pounds of dry ice in dry ice chamber on the day shipment is made.

iii. Container must reach destination within 15 days from date of shipment.

b. All diborane shipments except those which qualify as "empty" by subparagraph (c) must also comply with the following:

   i. Filling is to be by weight only.

   ii. Container must be precooled with liquid nitrogen to below minus 80°C. and excess nitrogen removed prior to charging with diborane.

c. Container shipped as "empty" of diborane must also comply with the following:

   i. Must be verified to be "empty" by one of the following:

      (a) The empty weight must not exceed the marked tare weight by more than 5 pounds, or

      (b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube.

   ii. Pressure must be vented to between 25 and 50 psig at the time container is emptied.

d. Provisions for shipment of methanol are as follows:

   i. The only time methanol may be shipped is to satisfy contractor's qualification acceptance tests. Under these conditions neither the transit time restrictions nor the requirement for precooling apply.

   ii. Additionally, the requirements of subparagraphs (a), (b), and (c) of this paragraph and the entire paragraph (9) do not apply to methanol shipments.

It was noted that this request did not intend any change in the terms of the permit.

Mr. Graziano, Director and Chief Inspector, Bureau of Explosives (AAR) replied to the request for authorization to ship quantities less than 100 pounds of diborane on a reduced time schedule. Their Chief Chemist, Charles Schultz, felt that some experimental measurements would be necessary before they could approve the request.

A letter was sent to DOT Office of Hazardous Materials on 28 March 1972 withdrawing the request for zero to 100-pound quantities, with the understanding that Callery or NASA could reactivate the request by submitting additional data (which NASA expected to obtain during use of the container). This letter also expedited action on rewording of the permit and ruling on rail express shipment.

Following a meeting with Mr. Grothe at the DOT-OHM Office on 13 April 1972, a letter was sent to DOT on 21 April 1972 reviewing all matters pending and introducing the need for incorporating into the permit some provision for shipment of recooled containers. The complete letter, included among the addenda in Appendix A, contained the formal request for shipment of recooled containers as follows:

> For shipment of a container recooled to a diborane pressure of 32 psig (corresponding to diborane temperature of -70°C.) to reach destination within
>
> 15 days for 175 to 200 lbs. B_2H_6
> 12 days for 100 to 174 lbs. B_2H_6

Detailed technical information was included in support of this request.

First Revision to DOT Special Permit No. 6522, dated 17 April 1972, was received on 24 April 1972; and contained all of the rewording changes except the provision for recooling. The additional changes in the revised permit to include shipment of recooled containers were outlined in Callery's letter of 24 April 1972.

In a letter dated 20 April 1972 DOT ruled that blanket approval could not be given for rail express shipment of diborane because of the long-standing prohibition by Bureau of Railroad Safety against shipments of Class A poisons by railway express in passenger train service. It was added that if the occasion arises, and the need is valid, authority may be granted for shipment on an individual basis.

Shipment of containers cooled to -70°C. (instead of -80°C) was discussed further with DOT, and on 30 August 1972 the request was resubmitted in writing. The modifications proposed therein were adopted by DOT in Second Revision to DOT Special Permit No. 6522, issued 14 September 1972.

The revisions to Special Permit No. 6522 which have been proposed and accepted* to date are included at the end of this section, following the original permit. Together these revisions extend the versatility permitted in the use of the shipping container developed under this contract.

*Included is the 3rd Rev. to D.O.T. S.P. 6522, requested by JPL, and issued October 20, 1972 (p. 101).

DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20590

SPECIAL PERMIT NO. 6522

This special permit is issued pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, to authorize shipments of a poisonous and compressed gas and a flammable liquid under conditions as prescribed herein. This permit does not relieve any shipper or carrier from compliance with any requirement of the DOT regulations, except as specifically provided for herein.

Standard special permit requirements and conditions relating to package markings, preparation of shipping papers, shipping experience reports, etc., are published in 49 CFR 171.6. These requirements are part of this special permit.

1. BASIS. June 14 and July 29, 1971 petition by Callery Chemical Company, Callery, Pa.

2. COMMODITY. Diborane or methanol (methyl alcohol).

3. PROPER SHIPPING NAME (49 CFR 172.5).

   a. For Diborane: Both "Compressed gas, n.o.s. (Flammable)" and "Poisonous gas, n.o.s.".

   b. For Methanol: "Methanol" or "Methyl alcohol".

4. REGULATION WAIVED. 49 CFR §§173.119, 173.304(a)(1) and 173.328.

5. AUTHORIZED SHIPPER. The petitioner identified above and its customers that are registered with this Board.

6. PACKAGING PRESCRIBED. In a specially designed 36-inch diameter spherical inner tank having a maximum working pressure of 500 psig containing not less than 100 pounds nor more than 200 pounds of product. Inner tank is surrounded (except for the dry ice chamber) by a 48-inch diameter cylindrical shell containing perlite insulation and which must contain not less than 108 pounds of dry ice. Container must be fabricated, and assembled in accordance with details included in CVI Corporation's drawings A458-5800 through A458-5821 on file with this Board. Except as otherwise provided herein, the container must be qualified and prepared for shipment in accordance with the petitioner's design and performance specifications on file with this Board.

7. SPECIAL PACKAGING REQUIREMENTS.

a. Filling is to be by weight only.

b. Container must be precooled with liquid nitrogen to below minus 80°C. and excess nitrogen removed prior to charging with diborane.

c. Container must be equipped with an audio-visual alarm to indicate temperature in excess of minus 35°C.

d. The only time methanol may be shipped is to satisfy contractor's qualification acceptance tests. Under these conditions neither the transit time restrictions nor the requirement for precooling apply. Additionally, the requirements of subparagraphs (e) and (f) of this paragraph and the entire paragraph (9) do not apply to methanol shipments.

e. Containers shipped as empty must comply with the following:

i. Reach destination within 15 days from time of emptying container.

ii. Be verified to be in "empty" condition by checking liquid opening to assure that only vapor is vented, or if weight determination is made, the empty weight must not exceed the marked tare weight by more than 5 pounds.

iii. Have not less than 108 pounds of dry ice in dry ice chamber.

f. In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows: "IF NOT DELIVERED BEFORE _____ CARRIER MUST ADVISE THE CALLERY CHEMICAL COMPANY, CALLERY, PENNSYLVANIA, ALSO THE BUREAU OF EXPLOSIVES, WASHINGTON, D.C., BY WIRE." The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date shipment is offered for transportation.

8. MODES OF TRANSPORTATION AUTHORIZED. Motor vehicle.

9. SPECIAL TRANSPORTATION REQUIREMENTS.

a. A copy of this permit, kept current, must be carried aboard each motor vehicle.

b. Shipments of diborane made under the terms of this permit must be delivered within 15 days from date of shipment.

c. Each shipping paper must show thereon, following the commodity description, the notation, appropriately executed:

DOT SPECIAL PERMIT NO. 6522
DATE OF SHIPMENT
IF NOT DELIVERED BEFORE ____ DAYS
CARRIER MUST ADVISE BUREAU OF EXPLOSIVES, WASHINGTON, D.C. BY WIRE.

d. Each shipper must require acknowledgement of receipt of shipment from consignee by wire, to be confirmed in writing, and must promptly notify the Bureau of Explosives (AAR) of any such shipment not received at destination within two days after shipment is due.

e. Any common carrier by motor vehicle transporting diborane under the terms of this permit must be specifically approved by the Federal Highway Administration.

10. REPORTING REQUIREMENTS. Any incident involving loss of contents of the package must be reported to this Board as soon as practicable.

11. EXPIRATION DATE. September 15, 1972.

Issued at Washington, D.C.: 31 AUG 1971 (DATE)

W. R. Fiste
For the Administrator
Federal Highway Administration

Address all inquiries to: Secretary, Hazardous Materials Regulations Board, U.S. Department of Transportation, Washington, D.C. 20590. Attention: Special Permits.

Dist: a, d,

DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20590

SPECIAL PERMIT NO. 6522
FIRST REVISION
(COMPLETE)

This special permit is issued pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, to authorize shipments of a poisonous and compressed gas; and a flammable liquid under conditions as prescribed herein. This permit does not relieve any shipper or carrier from compliance with any requirement of the DOT regulations, except as specifically provided for herein.

Standard special permit requirements and conditions relating to package markings, preparation of shipping papers, shipping experience reports, etc., are published in 49 CFR 171.6. These requirements are part of this special permit.

1. BASIS. March 15 and 28, 1972 petition by Callery Chemical Company, Callery, Pa.; March 20, 1972 petition by Jet Propulsion Laboratory, Pasadena, Calif.

2. COMMODITY. Diborane or methanol (methyl alcohol).

3. PROPER SHIPPING NAME (49 CFR 172.5).

a. For Diborane: Both "Compressed gas, n.o.s. (Flammable)" and "Poisonous gas, n.o.s.".

b. For Methanol: "Methanol" or "Methyl alcohol".

4. REGULATION WAIVED. 49 CFR §§173.119, 173.304(a)(1) and 173.328.

5. AUTHORIZED SHIPPER. The petitioners identified above and their customers who register their identity with and receive acknowledgement from this Board and have a copy of the special permit.

6. PACKAGING PRESCRIBED. In a specially designed 36-inch diameter spherical inner tank having a maximum working pressure of 500 psig containing not less than 100 pounds nor more than 200 pounds of product. Inner tank is surrounded (except for the dry ice chamber) by a 48-inch diameter cylindrical shell containing perlite insulation. Container must be equipped with an audio-visual alarm to indicate temperature above minus 35°C. Container must be fabricated and assembled

in accordance with details included in CVI Corporation's drawings A458-5800 through A458-5821 on file with this Board. Except as otherwise provided herein, the container must be qualified and prepared for shipment in accordance with the Callery's design and performance specifications on file with this Board.

7. SPECIAL PACKAGING REQUIREMENTS.

a. All diborane shipments, including those which qualify as "empty" by subparagraph (c), must comply with the following:

i. In addition to the flammable gas label and the poison gas label, each outside shipping container must bear a conspicuous label reading as follows: "IF NOT DELIVERED BEFORE ..... CARRIER MUST ADVISE (Insert name and address of shipper), ALSO THE BUREAU OF EXPLOSIVES, WASHINGTON, D.C., BY WIRE." The date inserted in the blank space on this label must not be in excess of the number of days prescribed herein from the date shipment is offered for transportation.

ii. Container must have not less than 108 pounds of dry ice in dry ice chamber on the day shipment is made.

iii. Container must reach destination within 15 days from date of shipment.

b. All diborane shipments except those which qualify as "empty" by subparagraph (c) must also comply with the following:

i. Filling is to be by weight only.

ii. Container must be precooled with liquid nitrogen to below minus 80°C. and excess nitrogen removed prior to charging with diborane.

c. Container shipped as "empty" of diborane must also comply with the following:

i. Must be verified to be "empty" by one of the following:

(a) The empty weight must not exceed the marked tare weight by more than 5 pounds, or

(b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube. Pressure should be vented to between 25 and 50 psig at the time container is emptied.

d. Provisions for shipment of methanol are as follows:

i. The only time methanol may be shipped is to satisfy contractor's qualification acceptance tests. Under these conditions neither the transit time restrictions nor the requirement for precooling apply.

ii. Additionally, the requirements of subparagraphs (a), (b), and (c) of this paragraph and the entire paragraph (9) do not apply to methanol shipments.

8. MODES OF TRANSPORTATION AUTHORIZED. Motor vehicle.

9. SPECIAL TRANSPORTATION REQUIREMENTS.

a. A copy of this permit, kept current, must be carried aboard each motor vehicle.

b. Shipments of diborane made under the terms of this permit must be delivered within 15 days from date of shipment.

c. Each shipping paper must show thereon, following the commodity description, the notation, appropriately executed:

DOT SPECIAL PERMIT NO. 6522
DATE OF SHIPMENT_____
IF NOT DELIVERED BEFORE_____
DAYS, CARRIER MUST ADVISE
BUREAU OF EXPLOSIVES, WASHINGTON,
D.C. BY WIRE.

d. Each shipper must require acknowledgement of receipt of shipment from consignee by wire, to be confirmed in writing, and must promptly notify the Bureau of Explosives (AAR) of any such shipment not received at destination within two days after shipment is due.

e. Any common carrier by motor vehicle transporting diborane under the terms of this permit must be specifically approved by the Federal Highway Administration.

10. REPORTING REQUIREMENTS. Any incident involving loss of contents of the package must be reported to this Board as soon as practicable.

11. EXPIRATION DATE. September 15, 1972.

Issued at Washington, D.C.:

for B.G. Milstead 17 APR 1972

W. R. Fiste (DATE)
For the Administrator
Federal Highway Administration

Address all inquiries to: Secretary, Hazardous Materials Regulations Board, U.S. Department of Transportation, Washington, D.C. 20590. Attention: Special Permits.

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DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20590

SPECIAL PERMIT NO. 6522
SECOND REVISION

Pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, and on the basis of the April 21 and August 30, 1972 petitions by Callery Chemical Co., Callery, Pennsylvania.

Special Permit No. 6522 is hereby amended by deleting subparagraph (7(a)(iii)); amending subparagraphs (7(b)(ii)), (7(c)(i)(b)), (9b) and paragraph (11); adding subparagraphs (7(b)(iii)) and (7(c)(ii) and (iii)) as follows:

"7a. * * *

i. * * *

ii. * * *

iii. Deleted.

"7b. * * *

i. * * *

ii. Containers precooled to minus 80°C. with liquid nitrogen must reach destination within 15 days from date of shipment. True temperature at or below -80°C. must be confirmed by pressure of 18 psig or lower on date of shipment.

iii. Containers cooled to minus 70°C. with dry ice (or liquid nitrogen) must reach destination within the following schedule.

(a) 15 days from date of shipment when diborane net weight is 175 to 200 pounds.

(b) 12 days from date of shipment when diborane net weight is 100 to 174 pounds.

True temperature at or below -70°C. must be confirmed by pressure of 32 psig or lower on date of shipment.

"7c. * * *

i. * * *

(a) * * *

(b) Level of liquid diborane must be below the bottom of the dip tube. Loss of liquid seal will be evident by ability to vent gas pressure from the container (to user's tank or other proper vent system) through the dip tube.

ii. Pressure should be vented to between 25 and 50 psig at the time container is emptied.

iii. Containers shipped as empty must reach destination within 15 days from date of shipment.

"9a. * * *

"9b. Shipment of diborane made under the terms of this permit must be delivered within the time prescribed in subparagraph 7(b).

"11. EXPIRATION DATE. September 15, 1973."

All other terms of this permit, as revised, remain unchanged. The complete permit currently in effect consists of the First and Second Revisions.

Issued at Washington, D.C.:

W. R. Fiste
For the Administrator
Federal Highway Administration

9-14-72
(DATE)

Address all inquiries to: Secretary, Hazardous Materials Regulations Board, U. S. Department of Transportation, Washington, D.C. 20590. Attention: Special Permits.

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DEPARTMENT OF TRANSPORTATION
HAZARDOUS MATERIALS REGULATIONS BOARD
WASHINGTON, D.C. 20590

SPECIAL PERMIT NO. 6522
THIRD REVISION

Pursuant to 49 CFR 170.15 of the Department of Transportation (DOT) Hazardous Materials Regulations, as amended, and on the basis of the September 22, 1972 petition by Jet Propulsion Laboratory, Pasadena, California:

Special Permit No. 6522 is hereby amended by adding paragraph (6a) to read as follows:

"6a. As an alternate to paragraph (6), shipments of diborane in quantities less than 100 pounds per container prescribed herein may be made subject to the following conditions:

i. The container and its contents must be precooled to minus 70°C. or lower, and the cooldown temperature must be confirmed by a measured ullage pressure of 32 psig or lower on the date of shipment.

ii. The container must have not less than 108 pounds of dry ice in the dry ice chamber on the day of shipment.

iii. The container must reach its destination within three days from the date of shipment.

iv. Shipments are limited to highway transportation via private carriage only.

v. All requirements of the permit, except as otherwise provided in this paragraph, are applicable.

All other terms of this permit, as revised, remain unchanged. The complete permit currently in effect consists of the First, Second and Third Revisions.

Issued at Washington, D.C.:

for B.J. Milster — 20 OCT 1972

W. R. Fiste
For the Administrator
Federal Highway Administration

(DATE)

Address all inquiries to: Secretary, Hazardous Materials Regulations Board, U.S. Department of Transportation, Washington, D.C. 20590. Attention: Special Permits.

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Jet Propulsion Laboratory, Pasadena, Ca.
North American Rockwell, Canoga Park, Ca.
Callery Chemical Co., Callery, Pa.

APPENDIX A

RECORD OF CONTACTS WITH D.O.T. REGARDING SPECIAL PERMIT FOR 200-POUND DIBORANE SHIPPING CONTAINER

[See VOLUME II]

APPENDIX B

OPERATING INSTRUCTIONS

DIBORANE SHIPPING CONTAINER

DOT SPECIAL PERMIT NO. 6522

[See VOLUME III]

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